GEORGIA INSTITUTE OF TECHNOLOGY
OFFICE OF CONTRACT ADMINISTRATION
SPONSORED PROJECT INITIATION

Date: 9/7/79

Project Title: Determination of Volume Solids of Paints and Coatings by Accurate Dry Film Thickness Measurement

Project No: A-2440

Project Director: Dr. C.J. Ray

Sponsor: Avondale Shipyard, Inc., New Orleans La. 70150

Agreement Period: From 8/1/79 Until 7/31/80

Type Agreement: P.O. N451 dated 7/27/79 (under Dept. of Commerce prime No. 5-38071)

Amount: $46,633

Reports Required: Monthly Letter Reports; Quarterly Progress Reports; Final Technical Report.

Sponsor Contact Person (s):

Technical Matters

Mr. John W. Peart
Project Director
Avondale Shipyards, Inc.
Mail Station #1H
New Orleans, Louisiana 70150
(504) 436-2121, ext. 494

Contractual Matters
(thru OCA)

Mr. S.L. Meredith
Avondale Shipyards, Inc.
P.O. Box 50280
New Orleans, Louisiana 70150

Defense Priority Rating: None

Assigned to: CMSL/MSD (~~School~~/Laboratory)

COPIES TO:

- Project Director
- Division Chief (EES)
- School/Laboratory Director
- Dean/Director—EES
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- Project File (OCA)
- Project Code (GTRI)
- Other

CA–3 (3/76)

GEORGIA INSTITUTE OF TECHNOLOGY
OFFICE OF CONTRACT ADMINISTRATION

SPONSORED PROJECT TERMINATION

Date: 4/29/81

Project Title: Determination of Volume Solids of Paints and Coatings by Accurate Dry Film Thickness Measurement

Project No: A-2440

Project Director: Mr. L. E. Henton

Sponsor: Avondale Shipyard, Inc., New Orleans La. 70150

Effective Termination Date: 11/30/80

Clearance of Accounting Charges: 11/30/80

Grant/Contract Closeout Actions Remaining:

x Final Invoice ~~and Closing Documents~~

_ Final Fiscal Report

_ Final Report of Inventions

X Govt. Property Inventory & Related Certificate

_ Classified Material Certificate

_ Other ______________

Assigned to: EMSL/MSD (~~School~~/Laboratory)

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Other: ______________

A-2440

ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

September 20, 1979

John Peart, Research Manager
Avondale Shipyards, Inc.
P. O. Box 50280
New Orleans, Louisiana 70150

Subject: The Determination of Volume Solids of Paints and Coatings By Accurate Dry Film Thickness Measurements. (Ga. Tech Project A-2440)

Dear John:

The first monthly progress report is attached covering the period of August 16 through August 31. The expenditures represent the time and effort Les Henton contributed to familiarize himself with the scope of the project and to set-up and organize a detailed plan to accomplish the tasks outlined in the proposal.

Sincerely,

Charles J. Ray

CJR:gp

Attachment

Monthly Progress Report

Number 1

Project A-2440

Determination of Volume Solids of Paints and Coatings By Accurate Dry Film Thickness Measurements

August 16 - August 31, 1979

Submitted to

Avondale Shipyards, Inc.

New Orleans, Louisiana 70150

by

Chemical and Material Sciences Laboratory

Engineering Experiment Station

Georgia Institute of Technology

Atlanta, Georgia 30332

Project Director

L. E. Henton

I. Work Progress

Task 1. Selection and Preparation of Coating Systems

A list of coatings representing the generic types of interest to the marine coatings industry has been requested of the Program Manager. These commercial coatings will be supplemented with other formulations suggested by coatings material suppliers to broaden the scope of the proposed volume solids technique as well as the present ASTM method.

Task 2. Volume Solids Determination

The current ASTM method D2697-73 has been reviewed. A list of materials and equipment needed to perform the test has been assembled so that the technical help can begin assembly and/or procurement as needed.

Availability and cost data on precision micrometers suitable for use per ASTM D-1005 are being collected. Other measurement techniques are also being explored such as beta-ray backscatter.

Task 3. Comparison of Methods

Work is not scheduled to start until the second reporting period.

Task 4. Standardization of Volume Solids Measurement

Work is not scheduled to start until the ninth month of the contract period.

II. Future Work

During the next reporting period it is planned to select the specific coating systems to be used in the program so that samples and/or the necessary materials can be procured. The equipment for the ASTM D2697-73 (volume nonvolatile matter) will be set-up and tested. The necessary supplies will be gotten. Precision film thickness standards will be procured so that the film thickness instruments can be accurately calibrated.

III. Budget

The following is a statement of the project budget based on information supplied by the Engineering Experiment Station accounting office.

	Personal Services	Retirement	Overhead	Travel	M&S	Total
Budget	$22,995.00	$2417.00	$17,476.00	$ 550	$1,695	$45,138.00*
Expended	$ 1,007.31	$ 105.87	$ 756.56			$ 1,878.74
Encumbered						
Free Balance	$21,987.69	$2311.13	$16,719.44	$ 550	$1,695	$43,263.26

* Excludes $1,500 for capital equipment.

A-2440

ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

October 10, 1979

John Peart, Research Manager
Avondale Shipyards, Inc.
P. O. Box 50280
New Orleans, Louisiana 70150

Subject: The Determination of Volume Solids of Paints and Coatings by Accurate Dry Film Thickness Measurements. (Ga. Tech Project A-2440)

Dear John:

Attached is the second Monthly Progress Report covering the period of September 1 through September 30.

I am having great difficulty in reaching you by telephone, to discuss certain matters concerning the project. If we have not conversed by the time you receive this letter, I would appreciate you giving me a call at 404-424-9651.

Thank you very much.

Sincerely,

Leslie E. Henton

LEH:gp

Attachment

Monthly Progress Report

Number 2

Project A-2440

Determination of Volume Solids of Paints and Coatings By Accurate Dry Film Thickness Measurements

September 1 - September 30, 1979

Submitted to

Avondale Shipyards, Inc.

New Orleans, Louisiana 70150

by

Chemical and Material Sciences Laboratory

Engineering Experiment Station

Georgia Institute of Technology

Atlanta, Georgia 30332

Project Director

L. E. Henton

I. Work Progress

Task 1. Selection and Preparation of Coating Systems

A list of coatings representing the generic types of interest to the marine coatings industry has been requested of the Program Manager. As of this date, we have not received it. However, we have solicited non-proprietary formulations from resin and pigment suppliers.

The raw materials required to make the formulations that we have received have been ordered, and we will proceed to make these formulations as quickly as possible.

Task 2. Volume Solids Determination

The necessary materials and equipment required to perform the current ASTM Method D2697-73 "Volume Nonvolatile Matter in Clear or Pigmented Coatings", has been procured and assembled. Precision film thickness standards have been ordered.

Task 3. Comparison of Methods

This work cannot be initiated until we have the approval of the program manager to obtain the following equipment: Precision Micrometers and a measuring Stand for the "Dual-Scope" Film Thickness Gauge, as the funds for obtaining this equipment will come from the capital equipment budget.

Task 4. Standardization of Volume Solids Measurement

Work is not scheduled to start until the ninth month of the contract period.

II. Future Work

During the next reporting period it is planned to select the specific coating systems to be used in the program so that samples and/or the necessary materials can be procured. Technical people will make test runs on determining volume solids according to ASTM D2697-73.

The equipment for testing film thicknesses by ASTM D1005-51, will have been received, set-up, and tested. This will now enable us to begin comparing the two methods.

III. Budget

The following is a statement of the project budget based on information supplied by the Engineering Experiment Station accounting office.

	Personal Services	Retirement	Overhead	Travel	M & S	Total
Budget	$ 22,995.00	$ 2,417.00	$ 17,476.00	$ 550.00	$1,695.00	$ 45,13
Expended	$ 2,665.38	$ 280.14	$ 2,025.69		$ .30	$ 4,97
Encumbered					$ 175.50	
Free Bal.	$ 20,329.62	$ 2,136.86	$ 15,450.31	$ 550.00	$1,519.20	$ 39,98

*Excludes $1,500 for capital equipment

A-2440

ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

December 5, 1979

John Peart, Research Manager
Avondale Shipyards, Inc.
P. O. Box 50280
New Orleans, Louisiana 70150

Subject: P. O. No. N-451, "Determination of Volume Solids of Paints and Coatings by Accurate Dry Film Thickness Measurements" (EES Project A-2440)

Dear John:

Attached is the third Monthly Progress Report covering the period of November 1 through November 30.

We are looking forward to your visit on December 12th. I would appreciate you bringing a list of your current paint suppliers.

Thanks very much.

Sincerely,

Leslie E. Henton

LEH:gp

Attachment(s)

Monthly Progress Report

Number # 3

Project A-2440

Determination of Volume Solids of Paints and Coatings By Accurate Dry Film Thickness Measurements

November 1 -- November 30, 1979

Submitted to

Avondale Shipyards, Inc.

New Orleans, Louisiana 70150

by

Chemical and Material Sciences Laboratory

Engineering Experiment Station

Georgia Institute of Technology

Atlanta, Georgia 30332

Project Director

L. E. Henton

I. WORK PROGRESS

TASK 1. Selection and Preparation of Coating Systems

A list of the coatings representing the selected generic types that we have selected was given in the first Quarterly Report. If there are any additional coatings the Program Manager wishes to have analyzed for volume solids, please inform us in order that they may be added to our program at this time.

TASK 2. Volume Solids Determination

Subtask 2.1 Volume Solids Determination by ASTM D 2697-73

We have analyzed 11 of the coatings selected in Task I for volume solids using the ASTM D 2697-73 method. This was done in quadruplicate and characterized by standard statistical techniques (e.g. average and standard deviations). The densities of the 11 coatings were also determined using the ASTM D-1475-60 method. The drying or conditioning of the films was done by the appropriate chemistry of the various generic types and the manufacturers' recommendations. The effect of forced drying/curing on the volume solids values is also being examined at this time.

Subtask 2.2 Volume Solids Determination by Precision Film Thickness Measurement

We have received the approval of the Program Manager to obtain the necessary equipment to perform this subtask. Purchase orders have been placed and we are waiting for delivery.

TASK 3. Comparison of Methods

This task initiation is dependent upon the implementation of Subtask 2.2.

TASK 4. Standardization of Volume Solids Measurements

This task is scheduled to be initiated in the ninth month of the contract period.

II. FUTURE WORK

During the next reporting period we will have received the necessary equipment to initiate Subtask 2.2 and TASK 3.

III. BUDGET

The following is a statement of the project budget based on the information supplied by the Engineering Experiment Station Accounting Office, for the period ending November 30, 1979.

	Personal Services	Retirement	Overhead	Travel	Materials & Supplies	TOTAL
Budget	$22,995.00	$2,417.00	$17,476.00	$550.00	$ 1,695.00	$45,133.00*
Expended	6,513.58	684.59	4,950.32		197.27	12,345.76
Free Balance	16,481.42	1,732.41	12,525.68	550.00	1,497.73	32,787.24

* Excludes $1,500.00 for capital equipment

Encumbered	677.50
	$ 822.50

~~File~~
A-244

ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

January 8, 1980

John Peart, Research Manager
Avondale Shipyards, Inc.
P. O. Box 50280
New Orleans, Louisiana 70150

Subject: P. O. No. N-451, "Determination of Volume Solids of Paints and Coatings by Accurate Dry Film Thickness Measurements", (EES Project A-2440)

Dear John:

Attached is the fourth Monthly Progress Report covering the period of December 1 through December 31.

I certainly appreciate you taking time out from your busy schedule to visit with Frank and me, to discuss our projects. Your comments were very informative and helpful, especially to my project. I am looking forward to receiving the other two methods of determining the volume solids for Inorganic Zinc-rich Coatings, and the International Ketimene cure epoxy samples.

Sincerely,

Leslie E. Henton
Chemical Material & Sciences Laboratory

LEH:gp

Monthly Progress Report

Number ~~3~~4

Project A-2440

Determination of Volume Solids of Paints and Coatings By Accurate Dry Film Thickness Measurements

December 1 - December 31, 1979

Submitted to

Avondale Shipyards, Inc.

New Orleans, Louisiana 70150

by

Chemical and Material Sciences Laboratory
Engineering Experiment Station
Georgia Institute of Technology
Atlanta, Georgia 30332

Project Director

L. E. Henton

I. WORK PROGRESS

TASK 1. Selection and Preparation of Coating Systems

In addition to the list of the selected coatings given in the first Quarterly Report, Mr. Peart will send us samples and data sheets of International's Ketimene cure epoxy series 400, which we will include in our test series. We are also contacting Marine coating suppliers requesting submission of their recommended products that would have a volume solid percentage that closely coincides with the results given by ASTM D-2697-73, to be used as a control and to also check the precision of both test methods.

Subtask 2.1 Volume Solids Determination by ASTM D-2697-73

As a result of the visit of Mr. John Peart, with us on December 12th and 13th, the following procedures will now be taken:

(1) For each of the generic type coatings, we will test volume solids for two (2) curing cycles:
   (a) As stated in the ASTM D-2697 method (3 hours at 105°C.)
   (b) The recommended curing cycle by the individual coating manufacturer, for application and curing of that particular coating.

(2) In any case, the method used for the weight non-volatile determination basically should follow the same curing schedule as used for the coated disks.

We have analyzed 14 of the coatings selected in Task 1 for volume solids using the ASTM D-2697-73 method. This was done in quadruplicate and characterized by standard statistical techniques (e.g. average and standard deviations). The densities of the 14 coatings were also determined using the ASTM D-1475-60 method.

Subtask 2.2 Volume Solids Determination by Precision Film Thickness Measurement

We have received the approval of the Program Manager to obtain the necessary equipment to perform this subtask. Purchase orders have been placed and we are waiting for delivery.

TASK 3. Comparison of Methods

This task initiation is dependent upon the implementation of Subtask 2.2.

TASK 4. Standardization of Volume Solids Measurements

This task is scheduled to be initiated in the ninth month of the contract period.

II. FUTURE WORK

During the next reporting period we will have received the necessary equipment to initiate Subtask 2.2 and TASK 3.

III. BUDGET

The following is a statement of the project budget based on the information supplied by the Engineering Experiment Station Accounting Office, for the period ending December 31, 1979.

	Personal Services	Retirement	Overhead	Travel	Materials & Supplies	TOTAL
Budget	$22,995.00	$2,417.00	$17,476.00	$550.00	$1,695.00	$45,133.00*
Expended	8,585.33	902.34	6,524.85	2.82	394.55	16,409.89
Free Balance	$14,409.67	$1,514.66	$10,951.15	$547.18	$1,300.45	$28,723.11

* Excludes $1,500.00 for capital equipment

Encumbered	677.50
	$ 822.50

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Monthly Letter Nos. 5 & 6

A-2440

ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

March 6, 1980

John Peart, Research Manager
Avondale Shipyards, Inc.
P. O. Box 50280
New Orleans, Louisiana 70150

Subject: P. O. No. N-451, "Determination of Volume Solids of Paints and Coatings by Accurate Dry Film Thickness Measurements", (EES Project A-2440)

Dear John:

Attached is the seventh monthly letter covering the period of February 1 through February 29.

We have now received the Sigma Coatings samples that we had listed as "in transit" on the list of generic coatings, in my letter of February 19. We are still looking forward to receiving the additional methods of determining the volume solids for Inorganic Zinc-rich coatings, and the International Ketimene-cure epoxy samples.

Sincerely,

Leslie E. Henton
Chemical & Material Sciences Laboratory

LEH:gp

Monthly Progress Report

Number 7

Project A-2440

Determination of Volume Solids of Paints and Coatings
Accurate Dry Film Thickness Measurements

February 1 - February 29, 1980

Submitted to

Avondale Shipyards, Inc.

New Orleans, Louisiana 70150

by

Chemical and Material Sciences Laboratory
Engineering Experiment Station
Georgia Institute of Technology
Atlanta, Georgia 30332

Project Director
L. E. Henton

I. Work Progress

Task 1. Selection and Preparation of Coating Systems

With the comments and input of Mr. John Peart, Program Manager, on our list of selected coatings in our letter of February 19, 1980, at least three coatings of each generic type will be selected for volume solids analysis as outlined in the statement of work. We shall then consider this task to be completed.

Task 2. Volume Solids Determination

Subtask 2.1 Volume Solids Determination by ASTM D 2697-73, "Standard Method of Test for Volume Non-Volatile Matter in Clear or Pigmented Coatings".

We have now completed the volume solids determinations by the ASTM D 2697-73 method, using two (2) curing cycles:

(1) as stated in the ASTM D 2697-73 method (3 hours at 105^{o}C),

(2) the recommended curing cycle by the individual coating supplier, for 90% of the coatings from the selected list. We have also completed the weight non-volatile determinations of the same coatings, following the same curing cycles as stated above.

Both the volume and weight non-volatile determinations were done in quadruplicate and characterized by standard statistical techniques (e.g. average and standard deviations). The densities of the above coatings were also determined using the ASTM D 1475-60 method.

Subtask 2.2 Volume Solids Determination by Precision Film Thickness Measurements

With the arrival of our precision film thickness measuring instruments, we are now beginning to analyze for volume solids of each of the coatings selected in Task 1, by the method outlined in our Statement of Work.

Subtask 2.3 Effect of Film Thickness on Volume Solids Valves

This task is now being implemented by determining the volume solids using the methods in Subtask 2.2, at two thickness levels, one greater than typically used and one less than typically used.

Subtask 2.4 Calculated Volume Solids

The selected list of coatings from Task 1, all have the manufacturer's stated volume solids on their supplied data sheets.

Task 3. Comparison of Methods

As volume solids data is generated in Task 2, we will begin to compare and analyze the merits of the two methods.

Task 4. Standardization of Volume Solids Measurements

This task is scheduled to be initiated in the ninth month of the contract period.

II. Future Work

To complete Subtask 2.1 and continue to analyze our selected list of coatings for volume solids using the precision film thickness measuring instruments as stated in Subtask 2.2.

III. Budget

The following is a statement of the project budget based on the information supplied by the Engineering Experiment Station Accounting Office for the period ending February 29, 1980.

	Personal Services	Retirement	Overhead	Travel	Material & Supplies	TOTAL
Budget	$ 22,995.00	$ 2,417.00	$ 17,476.00	$ 550.00	$1,695.00	$45,133.00*
Expended	12,308.94	1,293.52	9,354.79	2.82	441.44	23,401.51
Free Balance	10,686.06	1,123.48	8,121.21	547.18	1,253.56	21,731.49

* Excludes $1,500.00 for Capital Equipment

Encumbered	753.56
	$ 746.44

A -2440

ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

April 7, 1980

John Peart, Research Manager
Avondale Shipyards, Inc.
P.O. Box 50280
New Orleans, Louisiana 70150

Subject: P.O. No. N-451, "Determination of Volume Solids of Paints and Coatings by Accurate Dry Film Thickness Measurements", (EES Project A-2440)

Dear John:

Attached is the eighth monthly letter covering the period of March 1 through March 31.

There has been a modification of the list of coatings that we are examining. From the list in my letter of February 19, 1980 these changes are:

I. High-build Ketimine Cured Epoxy

2. International Paint Co. - Series 400 (to be received from John Peart)
3. Farboil - #99/99PR Primer replacing sigma
4. Farboil - #99/99E Topcoat coatings - samples

V. Chlorinated Rubber Coatings

3. Sigma Coatings - NUCOL CHRB-7311 (replacing Imperial Coatings Corp., Perma-Chlor #890)

VIII. Alkyd Coatings

3. Sigma Coatings Enamel 7240 (replacing Imperial Coatings Corp. - #88 Gloss Enamel)

IX. Inorganic Zinc-rich Coatings

3. Sigma Coatings - Tornusil MCF #7551 (replacing Imperial Coatings Corp. - Durazinc #555)

If these changes are agreeable to you, we shall consider this list to now be finalized.

Sincerely,

Leslie E. Henton
Chemical & Material Sciences Laboratory

LEH/pr

Monthly Progress Report

Number 8

Project A-2440

Determination of Volume Solids of Paints and Coatings

by Accurate Dry Film Thickness Measurements

March 1 - March 31, 1980

Submitted to

Avondale Shipyards, Inc.

New Orleans, Louisiana 70150

by

Chemical and Material Sciences Laboratory

Engineering Experiment Station

Georgia Institute of Technology

Atlanta, Georgia 30332

Project Director

L. E. Henton

I. Work Progress

Task 1. Selection and Preparation of Coating Systems

With the approval of Mr. John Peart, Program Manager of the modifications of our list of selected coatings in our letter of April 7, 1980, we shall consider this task to be completed.

Task 2. Volume Solids Determination

Subtask 2.1 Volume Solids Determination by ASTM D 2697-73. "Standard Method of Test for Volume Non-Volatile Matter in Clear or Pigmented Coatings".

We have now completed the volume solids determination by the ASTM D 2697-73 method, using two (2) curing cycles:

(1) as stated in the ASTM D 2697-73 method (3 hours at 105°C),

(2) the recommended curing cycle by the individual coating supplier, for 90% of the coatings from the selected list. We have also completed the weight non-volatile determinations of the same coatings, following the same curing cycles as stated above.

Both the volume and weight non-volatile determinations were done in quadruplicated and characterized by standard statistical techniques (e.g. average and standard deviations). The densities of the above coatings were also determined using the ASTM D 1475-60 method.

Subtask 2.2 Volume Solids Determination by Precision Film Thickness Measurements

We are now in the process of analyzing for volume solids using precision film thickness measurements and the method outlined in our statement of work of the selected coatings, that we have determined the volume solids of in subtask 2.1 using the ASTM D 2697-73 method.

Subtask 2.3 Effect of Film Thickness on Volume Solids Values

This task is now being implemented by determining the volume solids using the methods in Subtask 2.2, at two thickness levels, one greater than typically used and one less than typically used.

Subtask 2.4 Calculated Volume Solids

The selected list of coatings from Task 1, all have the manufacturer's stated volume solids on their supplied data sheets.

Task 4. Standardization of Volume Solids Measurements

This task is scheduled to be initiated next month (April, 1980).

II. Future Work

To complete Subtask 2.1 and continue to analyze our selected list of coatings for volume solids using the precision film thickness measuring instruments as stated in Subtask 2.2.

III. Budget

The following is a statement of the project budget based on the information supplies by the Engineering Experiment Station Accounting Office for the period ending March 31, 1980.

	Personal Services	Retirement	Overhead	Travel	Materials & Supplies	Total
Budget	$22,995.00	$2,417.00	$17,476.00	$550.00	$1,695.00	$45,133.00*
Expended	14,373.46	1,510.68	10,923.83	2.82	512.77	27,323.56
Free Balance	8,621.54	906.32	6,552.17	547.18	1,182.23	17,809.44

*Excludes $1,500.00 for capitol equipment
753.56
746.44

LIBRARY DOES NOT HAVE

Monthly Letter Nos. 9

A-244

ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

Phone: 404/424-9651

June 11, 1980

John Peart, Research Manager
Avondale Shipyards, Inc.
P.O. Box 50280
New Orleans, Louisiana 70150

Subject: P.O. No. N-451, "Determination of Volume Solids of Paints and Coatings by Accurate Dry Film Thickness Measurements", (EES Project A-2440)

Dear John:

Attached is the tenth monthly report covering the period of May 1 through May 31.

As you are aware of, a portion of our technician's work was stopped in May, at your request until some of our strategy concerning obtaining a more uniform film thickness could be worked out. I would like to express my appreciation for the guidance and assistance you gave us on your visit on May 20th, on this problem. After our phone discussion on May 28th, we are now pursuing the following directions:

1. We will measure the volume solids on two typical solvent type paints, (Sigma CRHB 7311 Chlorinated rubber and Imperial C-Flex #321 Vinyl), by the ASTM method using the Manufacturers Recommended Curing Time (MRCT). After making these determinations, we shall now heat the disks @ 150°C., monitoring the weight loss each day until constant weight is obtained. This will give us a curve for this determination. Now we will heat the same disks @ 200°C. until constant weight, giving us an additional curve.

2. Repeat the above (1.) using (NAPKO 7-2371, Epoxy-Amine Adduct and Deco-Rez DE-3490, Epoxy-Polyamide).

3. Using the same four paints above, determine the volume solids using the CMSL method, with ultra-sonic bath assistance in obtaining a uniform film thickness. We will try this with both water and trichloro-trifluoroethane as liquids in the bath.

4. I would also like to try the method of thoroughly mixing the appropriate solvent for each paint with the paint in a known ratio and metering out a known amount into the pans. Again

using the same four paints, it will probably save time in the mixing step, especially the epoxies.

However, my technician, Wayne Case, became ill on May 28th, requiring hospitalization. He will not return to work until June 16th. As I related to you in our phone conversation of May 28th, the preliminary results of using the ultra-sonic bath gave us a great deal better uniformity of film thicknesses and therefore much better precision in the volume solids film thickness method. I would like to pursue the above directions, taking a longer period of time (an additional month) with the same agreed upon project funds, with your permission.

If this is agreeable, I will initiate appropriate procedures through our Office of Contract Administration (OCA).

Sincerely,

Leslie E. Henton
Research Scientist II
Chemical & Material Sciences Laboratory

LEH:pr

Monthly Progress Report

Number 10

Project A-2440

Determination of Volume Solids of Paints and Coatings by Accurate Dry Film Thickness Measurements

May 1 - May 31, 1980

Submitted to

Avondale Shipyards, Inc.
New Orleans, Louisiana 70150

by

Chemical and Material Sciences Laboratory
Engineering Experiment Station
Georgia Institute of Technology
Atlanta, Georgia 30332

Project Director
L. E. Henton

I. Work Progress

Task 1. Selection and Preparation of Coating Systems

This task is completed.

Task 2. Volume Solids Determination

Subtask 2.1 Volume Solids Determination by ASTM D 2697-73. "Standard Method of Test for Volume Non-Volatile Matter in Clear or Pigmented Coatings".

We have now completed the volume solids determination by the ASTM D 2697-73 method, using two (2) curing cycles:

(1) as stated in the ASTM D 2697-73 method (3 hours at 105°C),

(2) the recommended curing cycle by the individual coating supplier, for 100% (28/28) of the coatings from the selected list. We have also completed the weight non-volatile determinations of the same coatings, following the same curing cycles as stated above.

Both the volume and weight non-volatile determinations were done in quadruplicated and characterized by standard statistical techniques (e.g. average and standard deviations). The densities of the above coatings were also determined using the ASTM D 1475-60 method.

Subtask 2.2 Volume Solids Determination by Precision Film Thickness Measurements

We were in the process of analyzing for volume solids using precision film thickness measurements and the method outlined in our statement of work of the selected coatings, that we have determined the volume solids of in subtask 2.1 using the ASTM D 2697-73 method. We had completed 46% (13/28) of the coatings from the selected list.

However, as a result of our difficulty in obtaining a sufficiently uniform dry film thickness, we will modify our procedure as outlined in steps no. 3 and no. 4 in the attached letter.

Subtask 2.3 Effect of Film Thickness on Volume Solids Values

This task is now being implemented by determining the volume solids using the methods in Subtask 2.2, at two thickness levels, one greater than typically used and one less than typically used.

Subtask 2.4 Calculated Volume Solids

The selected list of coatings from Task 1, all have the manufacturer's stated volume solids on their supplied data sheets.

Task 3. Comparison of Methods

The volume solids data generated in Task 2 will be compared and analyzed to assess the merits of the film thickness technique of determining volume solids against the present ASTM method. The comparison will be on the basis of the precision (reproducibility) of the two methods using standard statistical techniques. (e.g. standard deviation)

As a result of our previously stated difficulties, we will wait until the data has been generated by the modifications taken in Subtask 2.2 to make comparisons of the two methods.

Task 4. Standardization of Volume Solids Measurement

In order to initiate standardization of the better method (Task 3) we will complete the determinations for volume solids by both methods.

II. Future Work

To complete Subtask 2.2 and Task 3.

III. Budget

The following is a statement of the project budget based on the information supplied by the Engineering Experiment Station Accounting Office for the period ending May 31, 1980.

	Personal Services	Retirement	Overhead	Travel	Materials & Supplies	Total
Budget	$22,995.00	$2,417.00	$17,476.00	$550.00	$1,695.00	$45,133.00*
Expended	18,409.43	1,934.87	13,991.16	2.82	655.97	34,994.25
Free Balance	4,585.57	482.13	3,484.84	547.18	1,039.03	10,138.75

*Excludes $1,500.00 for capitol equipment
750.56
$ 749.44

A-244

ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

July 11, 1980

Mr. John Peart, Research Manager
Avondale Shipyards, Inc.
P.O. Box 50280
New Orleans, Louisiana 70150

Dear John:

Attached is the eleventh monthly report covering the period of June 1 through June 31.

In pursuing the new directions stated in our last monthly report, we found it was necessary to substitute Sigma Alkyd Enamel #7240 for Sigma Nucol CHRB 7311, because the chlorinated rubber paint begins to char and degrade at temperatures of 120°C and above. We also observed a strange phenomenon on heating the Imperial C-Flex #321 vinyl, the volume solid percentage increases as the temperature increases until it reaches a constant percentage. We are also substituting Carboline's 187 HFP amine cured epoxy for Napko's 7-2371, as we had trouble finding a suitable solvent. As previously stated using the ultra-sonic bath is giving us a much more uniform film thickness. However, we have found that water is the best liquid to use in the bath. Neither heavier or lighter than water liquids give results superior to water.

If it is agreeable with you, do you think it will be necessary to go through formal procedures to extend the time on this contract, with no additional extension of funds?

Sincerely,

Leslie E. Henton
Chemical & Material Sciences Laboratory

LEH:pr

Monthly Progress Report

Number 11

Project A-2440

Determination of Volume Solids of Paints and Coatings by Accurate Dry Film Thickness Measurements

June 1 - June 30, 1980

Submitted to

Avondale Shipyards, Inc.

New Orleans, Louisiana 70150

by

Chemical and Material Sciences Laboratory

Engineering Experiment Station

Georgia Institute of Technology

Atlanta, Georgia 30332

Project Director

L. E. Henton

I. Work Progress

Task 1. Selection and Preparation of Coating Systems

This task is completed.

Task 2. Volume Solids Determination

Subtask 2.1 Volume Solids Determination by ASTM D 2697-73. "Standard Method of Test of Volume Non-Volatile Matter in Clear or Pigmented Coatings".

We have now completed the volume solids determination by the ASTM D 2697-73 method, using two (2) curing cycles:

(1) as stated in the ASTM D 2697-73 method (3 hours at 105°C),

(2) the recommended curing cycle by the individual coating supplier, for 100% (28/28) of the coatings from the selected list. We have also completed the weight non-volatile determinations of the same coatings, following the same curing cycles as stated above.

Both the volume and weight non-volatile determinations were done in quadruplicate and characterized by standard statistical techniques (e.g. average and standard deviations). The densities of the above coatings were also determined using the ASTM D 1475-60 method.

Subtask 2.2 Volume Solids Determination by Precision Film Thickness Measurements

We had completed the volume solids determination for 46% (13/28) of the coatings from the selected list using precision film thickness measurements and the method outlined in our statement of work. However, as a result of our difficulty in obtaining a sufficiently uniform dry film thickness, we have modified our procedures.

Your suggestion of using the ultra-sonic bath has resulted in a much more uniform thickness. We will now attempt to complete our list of selected coatings using this method.

Subtask 2.3 Effect of Film Thickness on Volume Solids Values

This task is now being implemented by determining the volume solids using the methods in Subtask 2.2, at two thickness levels, one greater than typically used and one less than typically used.

Subtask 2.4 Calculated Volume Solids

The selected list of coatings from Task 1 all have the manufacturer's stated volume solids on their supplied data sheets.

Task 3. Comparison of Methods

The volume solids data generated in Task 2 will be compared and analyzed to assess the merits of the film thickness technique of determining volume solids against the present ASTM method.

As a result of our previously stated difficulties, we will wait until the data has been generated by the modifications taken in Subtask 2.2 to make comparisons of the two methods.

Task 4. Standardization of Volume Solids Measurements

In order to initiate standardization of the better method (Task 3) we will complete the determinations for volume solids by both methods.

II. Future Work

To complete Subtask 2.2 and Task 3 we will also measure the volume solids of two typical solvent type paints, by the ASTM method using the Manufacturers Recommended Curing Time (MRCT). After making these determinations we shall heat the disks @ 150°F, monitoring the weight loss each day until constant weight is obtained. This will give us a curve for this determination. Next we will heat the same disks @ 200°F until constant weight, giving us an additional curve.

Repeat the above using Carboline 187 HFP, Epoxy-Amine Adduct and Deco-Rez DE-3490, Epoxy Polyamide.

III. Budget

The following is a statement of the project budget based on the information supplied by the Engineering Experiment Station Accounting Office for the period ending June 30, 1980.

	Personal Services	Retirement	Overhead	Travel	Materials & Supplies	Total
Budget	$22,995.00	$2,417.00	$17,476.00	$550.00	$1,695.00	$45,133.00*
Expended	20,527.45	2,157.47	15,600.86	15.07	661.67	38,962.52
Free Balance	2,467.55	259.53	1,875.14	534.93	1,033.33	6,170.48

*Excludes	$1,500.00	for capital equipment
Expended	762.06	
Free Balance	737.94	

A-244

ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

August 14, 1980

John Peart, Research Manager
Avondale Shipyards, Inc.
P.O. Box 50280
New Orleans, Louisiana 70150

Dear John:

Attached is the twelfth monthly report covering the period of July 1 through July 31.

This report will indicate that the technical portion of this project is essentially finished. As we have discussed by telephone, I am enclosing some of the tables that will be presented in the final report, giving a somewhat overall picture of our findings. I would appreciate you examining these findings, so that you and I may discuss how this data should be presented in our final report. I would like to do this as quickly as possible, as our Budget Sheet indicates there is now only $3,123.13 remaining in our project funds. I would like to be able to finish writing the final report within the time-frame allowed by this figure (≃ September 15th). These funds ($3,123.13) will be used for my and our secretary's time, along with materials to finish writing the final report.

Sincerely,

Leslie E. Henton
Chemical & Material Sciences Laboratory

LEH:pr

Monthly Progress Report

Number 12

Project A-2440

Determination of Volume Solids of Paints and Coatings by Accurate Dry Film Thickness Measurements

July 1 - July 31, 1980

Submitted to

Avondale Shipyards, Inc.

New Orleans, Louisiana 70150

by

Chemical and Material Sciences Laboratory

Engineering Experiment Station

Georgia Institute of Technology

Atlanta, Georgia 30332

Project Director

L. E. Henton

I. Work Progress

Task 1. Selection and Preparation of Coating Systems

This task is completed.

Task 2. Volume Solids Determination

Subtask 2.1 Volume Solids Determination by ASTM D 2697-73. "Standard Method of Test of Volume Non-Volatile Matter in Clear or Pigmented Coatings".

We have now completed the volume solids determination by the ASTM D 2697-73 method, using two (2) curing cycles:

(1) as stated in the ASTM D 2697-73 method (3 hours at 105°C),

(2) the recommended curing cycle by the individual coating supplier, for 100% (28/28) of the coatings from the selected list. We have also completed the weight non-volatile determinations of the same coatings, following the same curing cycles as stated above.

Both the volume and weight non-volatile determinations were done in quadruplicate and characterized by standard statistical techniques (e.g. average and standard deviations). The densities of the above coatings were also determined using the ASTM D 1475-60 method.

Subtask 2.2 Volume Solids Determination by Precision Film Thickness Measurements

After modifying our procedures as outlined in our July Monthly Report, we have completed the volume solids determination for twenty-one (21) of the coatings from the selected list using precision film thickness measurements and the method outlined in our statement of work.

Subtask 2.3 Effect of Film Thickness on Volume Solids Values

This task was accomplished by determining the volume solids using the methods in Subtask 2.2, at two thickness levels, one greater than typically used and one less than typically used. Data will be presented in tabular form in the final report.

Subtask 2.4 Calculated Volume Solids

The selected list of coatings from Task 1 all have the manufacturer's stated volume solids on their supplied data sheets.

Task 3. Comparison of Methods

The volume solids data generated in Task 2 will be compared and analyzed to assess the merits of the film thickness technique of determining volume solids against the present ASTM method. (See enclosed table comparing volume solids data)

Task 4. Standardization of Volume Solids Measurements

In order to initiate standardization of the better method (Task 3) we have also measured the volume solids of two typical solvent type paints (Carboline - 3631HB and Carboline - 132), by the ASTM method using the Manufacturers Recommended Curing Time (MRCT). After making these determinations we heated the disks @ 150°F, monitoring the weight loss each day until constant weight was obtained. Next we heated the same disks @ 200°F until constant weight.

We repeated the above using Carboine 187 HFP, Epoxy-Amine Adduct and Matcote 1-844, Epoxy Polyamide. (See enclosed table comparing results) This data will give us a comparison of volume solids results determined by altering the ASTM D2697-73 method curing temperatures.

II. Budget

The following is a statement of the project budget based on the information supplied by the Engineering Experiment Station Accounting Office for the period ending July 31, 1980.

	Personal Services	Retirement	Overhead	Travel	Materials & Supplies	Total
Budget	$22,995.00	$2,417.00	$17,476.00	$550.00	$1,695.00	
	+ 833.70[1]	+ 92.63[1]	+ 608.60[1]	- 534.93[2]	-1,000.00[2]	
Total	$23,828.70	$2,509.63	$18,084.60	$ 15.07	$ 695.00	$45,133.00*
Expended	22,268.70	$2,350.92	$16,871.97	$15.07	$503.21	$42,009.87
Free Balance	1,560.00	158.71	$1,212.63	00.00	$191.79	$ 3,123.13

*Excludes	$1,500.00 for capital equipment
Expended	762.06
Free Balance	$ 737.94

1 - Transferred from (Travel and Material & Supplies)

2 - Transferred to (Personal Services, Retirement, and Overhead)

VOLUME SOLIDS COMPARISON DATA SHEET

SHEET 1

PAINT	*MVS %	ASTM-D 2697 3 hrs @ 105°C	Volume Solids *MRCT	Film Thickness Method-Vol. Solids Dial-Gauge Micrometer
I. High-Build Ketimine Cured Epoxy				
1. Farboil #99 E	87.0	73.5	76.7	61.1
2. Farboil #99 PR	87.0	76.3	85.0	66.7
II. Amine and Amine Adduct Cured Epoxy				
1. Carboline - 187HFP	66.0±1	68.6	73.8	78.1
2. Napko - 7-2371	44.3	55.6	57.8	43.3
3. Sigma - EHB - 7433	70.0	76.6	80.8	67.3
III. Polyamide Cured Epoxy				
1. Deco-Rez-DE-3490	40.0	45.2	44.1	40.0
2. Matcote Co. - 1-844	50.0±2.	63.7	67.2	57.1
3. Carboline - 193	50.0±1	56.4	57.3	56.4
VI. Vinyl Coatings				
1. Imperial Co.-#321 G-Flex	28±1	42.4	46.3	51.2
2. Sigma-NUCOL #7352	24.0	24.2	26.1	19.7
V. Chlorinated Rubber				
1. Carboline-3631HB	34±1	39.3	44.9	21.3
2. Sigma NUCOL-7311	47±1	49.9	53.5	44.4
VI. Urethanes				
1. Carboline - 132	55±1	56.3	55.8	47.3
2. Imperial-#1001	46.0	54.3	56.6	39.6
VII. Water-base Coatings				
1. International-Intertuf-XG921/XV1531	53.5	55.9	53.8	45.4
2. Sigma-7445	39.0	52.9	53.6	50.4
3. Porter-Epoxy 6610	36.4±2	41.7	42.7	34.1

VOLUME SOLIDS COMPARISON DATA SHEET

SHEET 2

	PAINT	MVS %	ASTM-D 2697 3 hrs @ 105^{o}C	Volume Solids MRCT	Film Thickness Method-Vol. Solids Dial-Gauge Micrometer
III.	Alkyd Coatings				
	1. Matecote-2400	35.±2	44.7		31.5
	2. Sigma - 7240-7000	50.0	41.0	44.1	23.7
IX.	Inorganic Zinc-rich Coatings				
	1. Matcote 1-289	63.9	63.9	63.9	70.5
	2. Sigma MCF-7551	56.0	74.7	74.2	74.5

*MVS - Manufacturer's Stated Volume Solids
MRCT - Manufacturer's Recommended Curing Time

COMPARISON OF VOLUME SOLIDS RESULTS BY
ALTERING THE ASTM D2697-73 METHOD
CURING TEMPERATURES

SHEET

PAINT	MVS*	MRCT**	3 HRS.@ 221°F Std. Temp.	150°F***	200°F***	Film Thickness Method
	%	%	%	%	%	%
Carboline 3631 HB	34±1	44.9	39.3	40.1	38.1	21.3
Carboline 132	55±1	55.8	56.3	53.1	48.8	45.8
Carboline 187HFP	66±1	73.9	68.6	71.7	70.7	78.1
Matcote 1-844	50±2	67.2	63.7	61.4	60.5	57.1

*MVS - Manufacturer's Stated Volume Solids

**MRCT - Manufacturer's Recommended Curing Temperature

*** - Samples Heated Until Constant Weight Obtained

A-2440

ENGINEERING EXPERIMENT STATION

GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

November 6, 1979

John Peart, Project Director
Avondale Shipyards, Inc.
P. O. Box 50280
New Orleans, Louisiana 70150

Subject: P. O. No. N-451, "Determination of Volume Solids of Paints and Coatings by Accurate Dry Film Thickness Measurements" (EES Project A-2440)

Dear John:

Attached is the first Quarterly Progress Report covering the period of August 16 through October 31.

I am looking forward to receiving your list of coatings currently being supplied and used at the Avondale Shipyards, to compare with our list of coatings "on-hand", and the coatings and formulations that we have requested from paint manufacturers and raw material suppliers, (lists included in Quarterly Progress Report). This will enable us to insure that we have at least three coatings of each generic type selected, that are representative of those used in the marine industry.

Sincerely,

Leslie E. Henton

LEH:gp

Enclosures -

cc: Mr. S. L. Meredith

QUARTERLY PROGRESS REPORT

Report Number 8 1

Project A-2440

Determination of Volume Solids of Paints and Coatings By Accurate Dry Film Thickness Measurements

August 16 - October 31, 1979

Submitted to

Avondale Shipyards, Inc.

New Orleans, Louisiana 70150

by

Chemical and Material Sciences Laboratory

Engineering Experiment Station

Georgia Institute of Technology

Atlanta, Georgia 30332

Project Director

L. E. Henton

I. WORK PROGRESS

Task 1. Selection and Preparation of Coating Systems

We have the following generic types of coatings "on hand" at EES:

Amine and Amine Adduct Cured Epoxy

Carboline 187 HFP

Polyamide Cured Epoxy

Carboline 190 HB
Carboline 193
Shell (DRH 151.3), Union Camp (Uni-Rez-2188) F125-25-1
Mil-P-24441/1 (Formula 150)
Mil-P-24441/2 (Formula 151) Mobile Paint Mfg. Co.
Mil-P-24441/5 (Formula 154)

Vinyl Coatings

Union Carbide Corp. - VP-3604 Hi-Build Vinyl Topcoat
Union Carbide Corp. - VP-3708 White Primer

Chlorinated Rubber Coatings

Carboline - 3631 HB

2 Component Urethanes

Carboline 132
Mobay - 255142

Water-Based Coatings

Ashland (108-1) White Topcoat - Acrylic Emulsion
Rohm & Haas (P-23-1) White Topcoat - Acrylic Latex
Ashland/Alcoa (PWB-23) Aluminum - Acrylic Emulsion Topcoat
Ashland/Reichard-Coulston (6A-2) Micaceous Iron Oxide/Acrylic Emulsion Topcoat
Rohm & Haas/Reichard-Coulston (6A-2) Micaceous Iron Oxide/Acrylic Emulsion Topcoat
Union Carbide/Reichard-Coulston (6A-2) Micaceous Iron Oxide/Acrylic Emulsion Topcoat
Carboline 288WB - Water-Based Epoxy
Carbo-Zinc #33 - Water-Base Inorganic Zinc Coating
Celanese-24-146 - Water-Based Polyamido-Amine Epoxy
Celanese-24-192 - Water-Based Polyamido-Amine Epoxy
Celanese-24-178 - Water-Based Amine Adduct Epoxy
Celanese-24-194 - Water-Based Amine Adduct Epoxy

Alkyd Coatings

Georgia DOT - 1B Orange Primer
Georgia DOT - 3A Green Topcoat
New York DOT - M-18HS Sage Green (708.11)
New York DOT - Gray Paint (708.10)
Carboline GP-10 Primer-Zn/Cr Alkyd
Carboline GP-62 Medium Oil Alkyd

Inorganic Zinc Rich Coatings

Union Carbide Corp. - MP-3825-ESP-X
Carbo-Zinc 11

We have contacted the following raw material suppliers, soliciting their assistance with raw materials and/or formulations:

High Build Ketimine Cured Epoxies

Shell Chemical Co.
Ciba-Geigy Corp.
R. T. Vanderbuilt Co., Inc.

Amine & Amine Adduct Cured Epoxies

Shell Chemcial Co.
Ciba-Geigy Corp.
R. T. Vanderbuilt Co., Inc.
Reichhold Chemicals, Inc.

Polyamide Cured Epoxies

Dow Chemical Co.
R. T. Vanderbuilt Co., Inc.

Vinyl Coatings

Union Carbide Corp.

Chlorinated Rubber Coatings

ICI Americas, Inc.

Urethanes - Two Component

Cargill, Inc.
Spencer-Kellogg, Division of Textron

Water-Based Coatings

Ashland Chemical Co.
Rohm & Haas Co.
Union Carbide Corp.

Alkyd Coatings

Ashland Chemical Co.
Reichhold Chemical Co.

Inorganic Zinc Rich Coatings

Union Carbide Corp.
New Jersey Zinc

We have also contacted, and have been promised finished paint products that are currently being supplied to the Marine Industry, by the following companies:

Ketamine Cured Epoxy	Matcote Co.	SICC(1)		
Amine & Amine Adduct Epoxy	Matcote Co.	SICC	SPV(2)	
Polyamide Cure Epoxy	Matcote Co.	SICC	SPV	Carboline
Vinyl Coatings	Matcote Co.	SICC	SPV	
Chlorinated Rubber Coatings	Matcote Co.	SICC	SPV	
2-Component Urethanes	Matcote Co.	SICC	SPV	
Water-Based Coatings	Matcote Co.	SICC	SPV	
Alkyd Coatings	Matcote Co.	SICC	SPV	Balt. Kopper
Inorganic Zinc Rich Coatings	Matcote Co.	SICC	SPV	

(1) Southern Imperial Coatings Corp.
(2) Standard Paint & Varnish Co.

Task 2. Volume Solids Determination

Subtask 2.1 Volume Solids Determination by ASTM D2697-73 "Standard Method of Test for Volume Non-Volatile Matter in Clear or Pigmented Coatings".

We have proceeded to determine volume solids on the coatings "on hand" by the ASTM D2697-73. We are also determining the effect of curing the coatings at the recommended drying or conditioning of the films versus the forced drying/curing on the volume solids values.

Subtask 2.2 Volume Solids Determination by Precision Film Thickness Measurement.

As was stated in September's Monthly Report, this subtask cannot be initiated until we have the approval of the program manager to obtain specific necessary equipment. This approval was requested through our GTRI Contract Administrator, Duane Hutchison, on October 15, 1979.

Subtask 2.3 Effect of Film Thickness on Volume Solids Values

This task is dependent upon the initiation of Subtask 2.2 and cannot be performed at this time.

Subtask 2.4 Calculated Volume Solids

The volume solids of the coating systems we have "on hand" are being calculated from the weight and density values of the components, along with the determination of the volume solids by the ASTM D2697-73 method.

Task 3. Comparison of Methods

Again, this task initiation is dependent upon initiation of Subtask 2.2.

Task 4. Standardization of Volume Solids Measurements

This work is not scheduled to start until the ninth month of the contract period.

II. Future Work

During the next reporting period, with the approval of the project manager, we will have purchased the necessary equipment to initiate Subtask 2.2 and we will be able to then initiate Task 3. Comparison of Methods.

III. Budget

The following is a statement of the project budget based on information supplied by the Engineering Experiment Station accounting office, for the period ending October 31, 1979.

	PERSONAL SERVICES	RETIREMENT	OVERHEAD	TRAVEL	MATERIALS & SUPPLIES	TOTAL
Budget	$ 22,995.00	$ 2,417.00	$ 17,476.00	$ 550.00	$ 1,695.00	$ 45,133.00*
Expended	$ 4,643.49	$ 488.04	$ 3,529.05		$ 187.52	$ 8,848.10
Encumbered						
Free Balance	$ 18,351.51	$ 1,928.96	$13,946.95	$ 550.00	$ 1,507.48	$ 36,284.90

* Excludes $1,500.00 for Capital Equipment
$36,284.90
$37,784.90 Actual Free Balance - October 31, 1979

A-2440

ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

February 7, 1980

John Peart, Research Manager
Avondale Shipyards, Inc.
P. O. Box 50280
New Orleans, Louisiana 70150

Subject: P. O. No. N-451, "Determination of Volume Solids of Paints and Coatings by Accurate Dry Film Thickness Measurements", (EES Project A-2440)

Dear John:

Attached is the second Quarterly Progress Report covering the period of November 1 through January 31.

The project is progressing approximately as scheduled. The only slight delay is in receiving the extremely accurate digital micrometer, for measuring the dry film thickness of the designated coatings. However, I do not perceive this to be a major problem, as delivery is expected shortly (the week of February 25th). We will still have sufficient time to complete all determinations as scheduled.

Sincerely,

Leslie E. Henton
Chemical Material & Sciences Laboratory

LEH:gp

Attachment

QUARTERLY PROGRESS REPORT

Number 6

Project A-2440

Determination of Volume Solids of Paints and Coatings By Accurate Dry Film Thickness Measurements

November 1 - January 31, 1980

Submitted to

Avondale Shipyards, Inc.

New Orleans, Louisiana 70150

by

Chemical and Material Sciences Laboratory

Engineering Experiment Station

Georgia Institute of Technology

Atlanta, Georgia 30332

Project Director

L. E. Henton

I. WORK PROGRESS

Task 1. Selection and Preparation of Coating Systems

In addition to the list of the selected coatings given in the first Quarterly Report, Mr. John Peart, Program Manager, is sending samples and data sheets of International Paint Company's ketimene cured epoxy series 400, which will also be included as part of our selected coatings. We have contacted several marine coating suppliers that have agreed to supply us with their recommended products that would have a volume solid percentage that closely coincides with the results given by using the ASTM-D-2693-73 method, to be used as a control, and also check the precision of both test methods.

Task 2. Volume Solids Determination

Subtask 2.1 Volume Solids Determination by ASTM D 2697-73, "Standard Method of Test For Volume Non-Volatile Matter in Clear or Pigmented Coatings".

We have now completed the volume solids determinations by the ASTM D 2697-73 method, using two (2) curing cycles:

(1) as stated in the ASTM D 2697-73 method (3 hours at 105°C),

(2) the recommended curing cycle by the individual coating supplier, for the application and curing of that particular coating,

for approximately 80% of the coatings from the selected list. We have also completed the weight non-volatile determinations of the same coatings, following the same curing cycles as stated above.

Both the volume and weight non-volatile determinations were done in quadruplicate and characterized by standard statistical techniques (e.g. average and standard deviations). The densities of the above coatings were also determined using the ASTM D 1475-60 method.

Subtask 2.2 Volume Solids Determination by Precision Film Thickness Measurement

The two instruments that are required to perform this determination were ordered on December 26, 1979. One, the measuring stand for our "Dual-Scope" eddy current film thickness measuring device has been received. The other instrument, a dial comperator (a dial indicator micrometer) is "in transit".

Task 3. Comparison of Methods

This task initiation is dependent upon the implementation of Subtask 2.2.

Task 4. Standardization of Volume Solids Measurments

This task is scheduled to be initiated in the ninth month of the contract period.

II. FUTURE WORK

During the next reporting period we will have received the necessary equipment to initiate Subtask 2.2 and Task 3.

III. BUDGET

The following is a statement of the budget based on the information supplied by the Engineering Experiment Station Accounting office, for the period ending January 31, 1980.

	Personal Services	Retirement	Overhead	Travel	Materials & Supplies	TOTAL
Budget	$22,995.00	$ 2,417.00	$17,476.00	$550.00	$ 1,695.00	$45,133.00 *
Expended	$10.491.40	$ 1,110.02	$ 8,026.66	$ 2.82	$ 415.35	$20,046.25
Free Balance	$12,503.60	$ 1,306.98	$ 9,449.34	$547.18	$ 1,279.65	$25,086.75

* Excludes $1,500.00 for Capital Equipment

Encumbered $ 677.50

$ 822.50

A-2440

ENGINEERING EXPERIMENT STATION

GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

May 9, 1980

John Peart, Research Manager
Avondale Shipyards, Inc.
P.O. Box 50280
New Orleans, Louisiana 70150

Subject: P.O. No. N-451, "Determination of Volume Solids of Paints and Coatings by Accurate Dry Film Thickness Measurements", (EES Project A-2440)

Dear John:

Attached is the Third Quarterly Progress Report covering the period of February 1 through April 30.

As stated in the Work Progress Report, Task 3. Comparison of Methods we are having difficulty obtaining a uniform dry film surface for our film thickness measurements. Any suggestions you may have will be greatly appreciated.

Sincerely,

Leslie E. Henton
Chemical & Material Sciences Laboratory

LEH:pr

QUARTERLY PROGRESS REPORT

Number 9

Project A-2440

Determination of Volume Solids of Paints and Coatings By Accurate Dry Film Thickness Measurements

February 1 - April 30, 1980

Submitted to

Avondale Shipyards, Inc.

New Orleans, Louisiana 70150

by

Chemical and Material Sciences Laboratory

Engineering Experiment Station

Georgia Institute of Technology

Atlanta, Georgia 30332

Project Director

L. E. Henton

I. Work Progress

Task 1. Selection and Preparation of Coating Systems

It was suggested by Mr. John Peart, Program Manager, that the selected coatings to be used in this project, would be finished paint products that are currently being supplied to the marine industry. Therefore with the approval of Mr. Peart, of the modifications of our list of selected coatings in our letter of April 7, 1980, and the arrival of the International Ketimene-cure epoxy samples, all the coating systems, will have been selected and collected. This will now complete this task. We will make determinations on 28 of these selected coatings, with a minimum of three different systems within each generic type.

Task 2. Volume Solids Determination

Subtask 2.1 Volume Solids Determination by ASTM D 2697-73. "Standard Method of Test for Volume Non-Volatile Matter in Clear of Pigmented Coatings".

We have now completed the volume solids determination by the ASTM D 2697-73 method, using two (2) curing cycles:

(1) as stated in the ASTM D 2697-73 method (3 hours at 105°C),

(2) the recommended curing cycle by the individual coating supplier, for 96% (27/28) of the coatings from the selected list. We have also completed the weight non-volatile determinations of the same coatings, following the same curing cycles as stated above.

Both the volume and weight non-volatile determinations were done in quadruplicated and characterized by standard statistical techniques (e.g. average and standard deviations). The densities of the above coatings were also determined using the ASTM D 1475-60 method.

Subtask 2.2 Volume Solids Determination by Precision Film Thickness Measurements

We are now in the process of analyzing for volume solids using precision film thickness measurements and the method outlined in our statement of work of the selected coatings, that we have determined the volume solids of in subtask 2.1 using the ASTM D 2697-73 method. We have now completed 46% (13/28) of the coatings from the selected list.

Subtask 2.3 Effect of Film Thickness on Volume Solids Values

This task is now being implemented by determining the volume solids using the methods in Subtask 2.2, at two thickness levels, one greater than typically used and one less than typically used.

Subtask 2.4 Calculated Volume Solids

The selected list of coatings from Task 1, all have the manufacturer's stated volume solids on their supplied data sheets.

Task 3. Comparison of Methods

The volume solids data generated in Task 2 will be compared and analyzed to assess the merits of the film thickness technique of determining volume solids against the present ASTM method. The comparison will be on the basis of the precision (reproducibility) of the two methods using standard statistical techniques. (e.g. standard deviation)

Based upon standard deviation calculations for the 13 coatings that have been analyzed for volume solids by both techniques, the ASTM D 2697-73 method is the more precise technique, with a tie 46% (6/13) between the two curing cycles, with one system (amine adduct-Napko #7-2371) having identical standard deviations. We have also observed that the precision or standard deviation using the manufacturer's recommended curing time for two water-borne systems is much better than that observed for the 3 hours @ 105°C curing cycle.

The lack of uniformity of the dry film surface is the contributing factor in the film thickness method not producing volume solid determinations that are as precise as the ASTM determinations. We have increased the number of film thickness measurements from three to eight and in some determinations to ten in an attempt to minimize this effect. We will continue to try various methods to improve our techniques in obtaining a more uniform surface for the remainder of the coatings.

Task 4. Standardization of Volume Solids Measurement

In order to initiate standardization of the better method (Task 3) we will complete all the determinations for volume solids by both methods on all the coatings on the select list.

II. Future Work

To complete Subtask 2.2 and Task 3.

III. Budget

The following is a statement of the project budget based on the information supplied by the Engineering Experiment Station Accounting Office for the period ending April 30, 1980.

	Personal Services	Retirement	Overhead	Travel	Materials & Supplies	Total
Budget	$22,995.00	$2,417.00	$17,476.00	$550.00	$1,695.00	$45,133.00*
Expended	16,113.94	1,693.61	12,246.59	2.82	623.84	30,680.80
Free Balance	6,881.06	723.39	5,229.41	547.18	1,071.16	14,452.20

*Excludes $1,500.00 for capitol equipment

	$1,500.00
	750.56
$	749.44

Final Report

Project No. A-2440

DETERMINATION OF VOLUME SOLIDS OF PAINTS AND COATINGS BY ACCURATE DRY FILM THICKNESS MEASUREMENTS

By

Leslie E. Henton,
GEORGIA INSTITUTE OF TECHNOLOGY
Engineering Experiment Station
Atlanta, Georgia 30332

March 1981

August 1, 1979 to November 30, 1980

Purchase Order No. N-451

Prepared for

AVONDALE SHIPYARDS, INC.
P. O. Box 50280
New Orleans, Louisiana 70150

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FOREWORD

Avondale Shipyards, Inc. has been contracted by the U.S. Department of Commerce, Contract No. 5-38071, to manage its research and development efforts in the area of surface preparation and coating.

Pursuant with this responsibility, the following research and development was sub-contracted to the Engineering Experiment Station, Georgia Institute of Technology.

"Development of a Standard Procedure for Determining Volume Solids of Coatings."

This research project was undertaken with the primary objective as the development of a standard analytical procedure to determine the volume solids of liquid coatings. The volume solids obtained was to accurately represent the volume of dried coating film (coverage) obtained from a gallon of liquid material.

Successful completion of the work would, at least, provide a detailed procedure for measuring the volume solids of coatings used in the marine industry. Once these procedures are adopted by the marine industry, coating suppliers can be required to report the volume solids on that basis and not values calculated from a formula sheet. This would help in estimating the quantity of paint required for various jobs, minimizing the probability of purchasing too little or too much paint with obvious dollar savings. With better volume solids data, the estimate of painting time can also be made more accurately using, for example, the effective solids output parameter promoted by Ginsberg.[1]

Mr. Leslie E. Henton, of the Engineering Experiment Station, served as Principal Investigator. Mr. Wayne Case, of the same institute, performed

all testing operations. On behalf of Avondale Shipyards, Inc., Mr. John Peart was the R & D Project Manager responsible for technical direction, editing, and publication of this report.

Special thanks are given to Mr. David Hurst, of the Engineering Experiment Station, Georgia Institute of Technology, for the film thickness measurement concept and to Mr. W. R. Tooke, Jr. of Micro-Metrics Company for supplying data from an ASTM round robin on dry film measurements. Also, we wish to acknowledge the contributions of the following corporations:

Avondale shipyards, Inc., New Orleans, Louisiana
Carboline Marine Corporation, St. Louis, Missouri
Farboil Company, Baltimore, Maryland
General Polymers Corporation, Cincinnati, Ohio
Imperial Coatings Corporation, New Orleans, Louisiana
International Paint Company, Inc., Union, New Jersey
Jotun-Baltimore Copper Paint Company, Baltimore, Maryland
Matcote Company, Inc., Houston, Texas
NAPCO Corporation, Houston, Texas
Porter Coatings, Louisville, Kentucky
Sigma Coatings, Harvey, Louisiana

EXECUTIVE SUMMARY

A new method to determine the volume solids of paints and coatings based on the measurement of dried film thickness over a known area has been studied in this work. It was compared to the American Society for Testing and Materials Method D 2697-73 Volume Nonvolatile Matter in Clear and Pigmented Coatings. This method determines the volume of the dry film by application of the Archimedes buoyancy effect. In addition, the project was structured to extend the ASTM method to coatings systems used in the marine industry.

The volume solids of several typical marine coating systems were determined using the proposed film thickness method as well as the current ASTM method. The type of coatings examined were ketimine cured epoxies, amine and amine adduct cured epoxies, polyamide cured epoxies, vinlys, chlorinated rubbers, alkyds, inorganic zinc-rich coatings, urethanes, and waterborne coatings. The film drying or curing conditions used were appropriate to the chemistry involved in the film forming process.

The results indicate that the precision of the ASTM method is better than the precision of the film thickness method. This is primarily due to poor film thickness uniformity. It was also shown that there is no benefit in time savings and sample handling in making volume solids determinations by the film thickness technique, when the manufacturer's recommended conditioning schedule is used to cure the paint film. The ASTM Method, then is the preferred one.

From the results of the work on this project, it is concluded that the user and the paint supplier must agree upon the curing or conditioning schedule, as the curing conditions can affect the volume solids values obtained.

Any further work done in pursuing the film thickness technique should be in the direction of obtaining a method that will give samples with uniform film thickness.

CONCLUSIONS

1. The precision in the film thickness method is much less than the precision of the ASTM method. This is due to the lack of film thickness uniformity.
2. There is no benefit in time savings and sample handling in making a volume solids determination by the film thickness method in comparison with the ASTM method.
3. The curing conditions can affect the volume solids values obtained so it is imperative that the manufacturer and user agree upon the conditioning schedule. This is already recognized in the ASTM method.
4. Although there were cases where the volume solids values obtained by the two methods agreed in terms of the student's t-test, the large variances in the film thickness method may negate the validity of those agreements.

2. PROJECT PLAN OF ACTION AND RESULTS

2.1 Objective

The objective was development of a standard analytical procedure for determining the volume solids of liquid coatings. The volume solids obtained was to accurately represent the volume of dried coating film (coverage) obtained from a gallon of liquid material.

2.2 General Approach

The present ASTM method for the determination of the volume solids of clear and pigmented coatings, ASTM D 2697-73,[2] is based on the indirect measurement of the volume of a dried paint film using the Archimedes buoyancy effect. The weight of the paint film, supported on a metal substrate, is determined in air and in some liquid of known specific gravity. The weight (mass) difference divided by the specific gravity of the liquid gives the volume of the paint film. This data in combination with the weight solids and the specific gravity of the wet paint is then used to calculate the volume solids. In principal, this method is highly accurate since it is based on well established gravimetric techniques. The method, however, is not used widely in the coatings industry. Volume solids typically are calculated from formulations or batch sheets using the density of the individual components and assuming that the volumes are additive. This assumption is, in general, incorrect. Hence, experimental volume solids and calculated volume solids will be different; the magnitude of this difference will be dependent on the magnitude of the error in assuming that the volumes are additive.

There are sources for error or differing interpretations to the application of ASTM D 2697-73 to the wide range of paints and coatings found in industry. One must determine if, for example, voids or pores are a proper part of the final film structure for, if so, a liquid must be used that will not penetrate into these voids. The displacement liquids used must also not be absorbed into the paint film, at least in the time it takes to make the weight measurements. Reasonable, intelligent modifications to the method must also be made based on the chemistry involved in the film forming process for each coating tested. Here, a particularly sensitive point is the conditioning procedures for obtaining a final dried film and the determination of the weight non-volatiles of the coating. The current method recommends drying for three hours at 105°C although this is qualified by a note which identifies the best drying conditions as those recommended by the manufacturer of the coating and similar to the in-use curing conditions. Unintentional abuses of the drying procedure have occurred. For example, a coating based on unsaturated polyester cured or crosslinked by in-situ, room temperature polymerization with styrene was subjected to the 105°C heating.[3] This, of course, volatilized the styrene, a normal component of the dried coating, which lead to completely erroneous results. Similar problems can be expected in systems that use low molecular weight materials that are crosslinked into the final film by reaction with absorbed water vapor such as urethane systems or ketimine-epoxy systems. It seems obvious, at this point, that for the wide range of coatings used in the marine industry appropriate methods of film drying or curing for volume solids measurements must be examined and developed.

2.2.1. New, Proposed Method

A method to determine the volume solids of paints and coatings based on the measurement of dried film thickness has been studied in this work. The method does not require the selection of a displacement liquid so that errors due to the penetration or non-penetration of the liquid into pores and/or voids and the swelling/absorption properties of the coating-liquid system are avoided.

The new method entails the measurement of the weight of the wet, freshly applied coating, the specific gravity of the wet coating, and its dry film thickness over a known area. Drying or curing conditions are selected appropriate to the coating system.

The volume solids, ϕ, of a paint or coating in this alternate method is given by

$$\phi = A \tau \rho / W \tag{1}$$

where A is the area of the film, τ is its thickness, ρ is the density of the wet paint, and W is the weight of the wet coating applied to area A. That is, the initial volume of paint applied is

$$V_i = W/\rho \tag{2}$$

and the final volume is

$$V_f = A\tau \tag{3}$$

In any experimental determination of a quantity, there are errors in the measurements which introduce uncertainties into the final, calculated value. The error analysis of the film thickness measurement technique performed here is based on a standard propagation of errors approach.[4] It represents the largest error in the volume solids one can reasonably expect.

The limit of error in the volume solids, $\lambda(\phi)$ is given by

$$\lambda(\phi) = \frac{A\tau}{W}\lambda(\rho) + \frac{A\rho\lambda(\tau)}{W} + \frac{\tau\rho}{W}\lambda(A) + \frac{A\tau\rho}{W^2}\lambda(W) \tag{4}$$

where $\lambda(\rho)$, $\lambda(\tau)$, $\lambda(A)$, and $\lambda(W)$ represent the limit of error for each of the measured quantities. On a relative basis, the limit of error is

$$\frac{\lambda(\phi)}{\phi} = \frac{\lambda(\rho)}{\rho} + \frac{\lambda(\tau)}{\tau} + \frac{\lambda(A)}{A} + \frac{\lambda(W)}{W} \tag{5}$$

i.e., the relative limit of error of the volume solids is equal to the sum of the relative limit of error for each experimentally determined quantity.

The limit of error in the density, $\lambda(\rho)$, can be taken as the limit specified in ASTM D 1475 Density of Paint, Varnish, Lacquer and Related Products since this method is used.[5] The value of the limit is 0.015 lb/gal. (0.002 kg/L) which represents 3σ (σ is the variance) limits. Using the density of water as 8.33 lb/gal as a reference point, the relative error limit is $\sim$0.2 per cent. Since most pigmented paints will have a density greater than that of water, the relative error value of 0.2 per cent is probably an upper limit.

The relative error in the weight of the wet sample is expected to be extremely small since the weight, determined to a tenth of a milligram (0.1 mg.), is on the order of 100 mg. This gives a relative error on the order of 0.1 per cent. This error, of course, may be larger depending on the volatility of the solvent blend in any particular paint. If rapid weight loss is a problem, it can be easily minimized by dispensing the wet sample from a syringe as cited in ASTM D 2369-73 Volatile Content of Paints.[6] The weight of wet paint deposited is then determined by the weight change of the syringe.

The errors associated with the area of the dish holding the wet and dry paint can be made small by using evaporating dishes which are constructed of aluminum and have smooth, flat bottom and nearly vertical sides.[7] If the nominal 50 mm diameter is accurate to 0.1 mm, the relative error becomes

$$\frac{\lambda(A)}{A} = \frac{0.2\text{mm}}{50\text{ mm}} = 0.004 \qquad (6)$$

Again, one can reasonably expect a maximum error on the order of a few tenths of a per cent.

The accurate measurement of film thickness is the critical part of the proposed approach to volume solids measurements. For the factors briefly explored above, the cumulative, relative error is 1 per cent as a maximum. For a first look at the film thickness precision, one can use data supplied through ASTM methods of film thickness measurement. If a non-magnetic sample cup is used, an instrument based on eddy currents induced in the substrate metal can be used. From an ASTM D 1400-67 round robin testing with eddy current instruments, the standard deviation between results from different laboratories was 0.11 mil.[8] If 1.96σ is taken as the limit for the error, (95 per cent confidence limits), the limit of relative error is

$$\frac{\lambda(\tau)}{\tau} \simeq \frac{0.22}{3} \simeq 0.072 \qquad (7)$$

using 3 mils as a typical film thickness.

It should be noted that the "in lab" standard deviation was 0.055

mil; this reduces the error limit to ~4 per cent in equation (7).

Keane and Shoemaker have reported on film thickness measurements for coatings on structural steel using various magnetic gages.[9] They conclude that the instruments are inherently accurate to within 15 per cent of the true thickness and that the accuracy is improved by several thickness determinations and averaging. This can also be seen in data reported below.

Table 1 contains the analysis of film thickness measurements using two different, commercial magnetic gages. This data was supplied by W. R. Tooke, Jr. of the Micro-Metrics Company.[10] In Table 1, the average film thickness, the per cent error defined as

$$\frac{\Delta\tau}{\tau} = \frac{\tau_{measured} - \tau_{shim}}{\tau_{shim}} \times 100 \qquad (8)$$

and the per cent relative error limit defined as

$$\frac{\lambda(\tau)}{\bar{\bar{\tau}}} = \frac{1.96\sigma}{\bar{\bar{\tau}}} \times 100 \qquad (9)$$

are reported. The limit of error is taken as 1.96σ ; for a normal distribution this represents the 95 per cent confidence limit.

Table I. Precision and Accuracy of Some Film Thickness Measurements

Instrument	Nominal Film Thickness (mil)	Average Film Thickness (mil)	$\lambda(\tau)$(mil)	$\frac{\Delta\tau}{\tau}$ (%)	$\frac{\lambda \tau}{\bar{\tau}}$(%)
Zorelco[a] 747-F	3.00	3.3	0.4	10	12
	4.73	5.0	0.4	5.7	8.4
	9.77	10.2	1.6	4.4	15
Zorelco 747-NF	3.00	2.9	0.5	3.3	17
	4.73	4.7	1.3	-0.6	28
	9.77	9.7	1.5	-0.7	15
Verimeter[b]	3.00	3.4	0.3	13	8.8
	4.73	5.3	0.7	12	13
	7.70	8.2	0.4	6.5	4.9

a Zorelco Ltd., P. O. Box 4444, Cleveland, Ohio 44125; tel. 216-441-6100
b Micro-Metrics Company, P. O. Box 13804, Atlanta, Georgia 30324; tel. 404-325-3243.

In general, the accuracy is better than the precision for this set of data. It is felt that this is due to a small sample size and different calibration standards used in generating the data. The average thickness is for four measurements but these four are two sets of duplicates only with each duplicate set measured after calibration with different standards. The precision of the thickness measurements is better exemplified by the analysis presented in Table 2. The data therein are presented as:

$$\bar{\tau} \quad \pm\lambda(\tau)(\text{mil})/\lambda(\tau)/\bar{\tau}(\%)$$

Table 2. Precision of Some Film Thickness Measurements

Instrument	Panel Area 1	Panel Area 2	Panel Area 3
Minitector[a]	6.3±0.1/2.3	16.3±0.7/4.3	1.2±0.2/17
Verimeter[b]	6.1±0.1/1.6	15.7±0.5/3.2	1.3±0.2/15

a Zormco Electronics Corporation, 8520 Garfield Blvd., Cleveland, Ohio 44125; telephone: 216-441-6100

b Micro-Metrics Company, P. O. Box 13804, Atlanta, Georgia 30324; telephone: 404-325-3243.

The data supplied by W. R. Tooke, Jr. of Micro-Metrics Company, was part of an ASTM round robin on dry film measurement. The average value reported is for six film thickness values measured in sets of three on two consecutive days. The precision is much better than that reported in Table 1, reflecting a better sample size and better calibrating procedures. The higher relative error for thin film, i.e., those ~1 mil, reflect the greater difficulty in determining the thickness of thin films. The absolute limit of error is still small: 0.2 mil. Since most marine coatings are used at thicknesses closer to the 6 and 16 mil figures of Table 2, it seems reasonable to use their limits of error in the total error analysis.

From the above, it is concluded that the limit of relative error the film thickness measurements using magnetic and eddy-current gages is on the order of 5-7 per cent. Hence, the final estimate of the limit of error for volume solids determined by accurate film thickness measurements is less than 10 per cent.

The precision or reproducibility of the present ASTM method is given as ±1.6 per cent absolute if water was the displaced fluid and ±3.9 per cent absolute if a hydrocarbon solvent is used.[2] These are values for agreement between the average of duplicate measurements in different laboratories. The relative limits depend, obviously, on the volume solids of the paint. None of the paints used in developing ASTM D 2697-73 were of the newer, high solids variety so the volume solids were most likely below 50 per cent and quite possibly below 40 per cent. On a relative basis then, the expected precision is in the range of 3-10 per cent. Hence, it appeared that the film thickness approach had merit and should be pursued in more detail. In addition, it was felt that the film thickness approach may be more convenient and rapid than the ASTM method since the measurement of film thickness is fast and eliminates errors due to various chemical and physical interactions between the film and the displacement fluid.

2.3 Plan of Action

2.3.1. Scope of Work

The volume solids of several types of coating systems used in the marine industry were to be determined using the proposed film measurement technique as well as the current ASTM method. The type of coatings to be examined were to include high build ketimine cured epoxies, amine and amine adduct cured epoxies, polyamide cured epoxies, vinyl based coatings, chlorinated rubber based coatings, alkyds, inorganic zincs, urethanes, and water-based coatings. Film drying or curing conditions used were to be appropriate to the chemistry involved in the film forming process. For example, the ketimine cured epoxies would be conditioned for seven days at standard conditions of 50±5 per cent relative humidity and 23±1 C. In total, the work was to extend the ASTM procedure to systems not used in its development and also allow a detailed, critical examination of the film thickness measurement approach.

The major steps in the program are presented in schematic form in

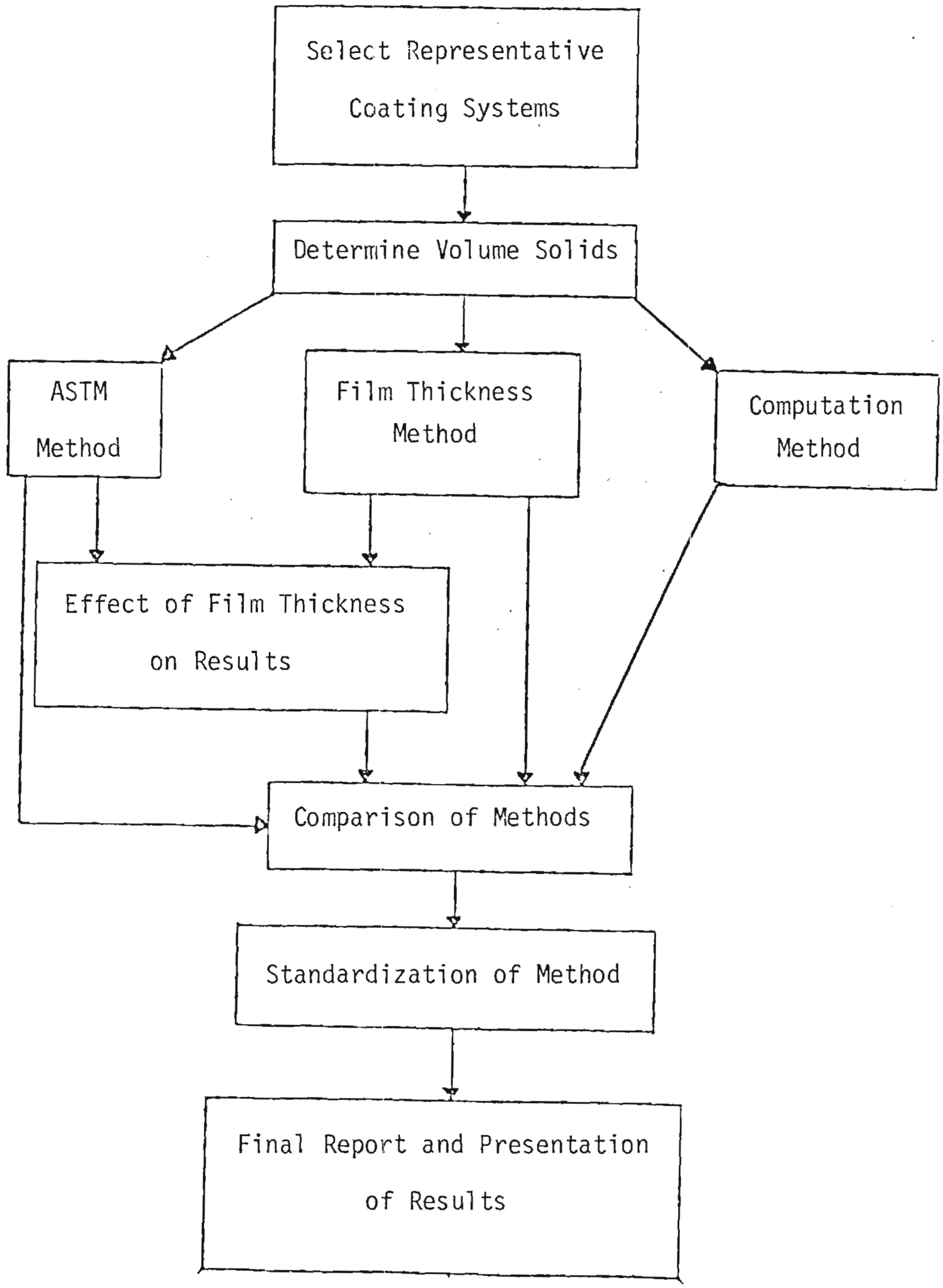

Figure 1. Schematic representation of the research plan.

Figure 1.

2.3.2. Statement of Work

In order to achieve the program objectives outlined above, the tasks were:

TASK 1. Selection and Preparation of Coating Systems

The generic type of coatings to be explored in the program have been designated in 2.3.1. Scope of Work.

At least two coatings of each generic type were to be selected with consultation with the Program Manager for volume solids analysis. This was done to insure that the coatings were representative of those used in the marine industry.

The selected coatings that were used in this project were commercial paint products currently being supplied to the marine industry.

TASK 2. Volume Solids Determination

Subtask 2.1 Volume Solids Determination by ASTM D-2697-73 Standard Method of Test for Volume Nonvolatile Matter in Clear or Pigmented Coatings.

Analyze each coating selected in Task 1 for volume solids according to ASTM D 2697-73. The values were based on at least four measurements, and characterized by standard statistical techniques (e.g. mean and standard deviation). The density of the wet paint was also determined according to ASTM D 1475-60 Standard Method of Test for Density of Paint, Varnish, Lacquer, and Related Products. The films were dried or conditioned with respect to the appropriate chemistry of the materials and as close to field use conditions as practicable. The approximate film thickness or weight was representative of field use thickness, as recommended by the paint manufacturer. The effect of forced drying/curing on the volume solids value was also explored using the procedure in ASTM D 2697-73.

Subtask 2.2 Volume Solids Determination by Precision Film Thickness Measurement.

Each of the coatings selected in Task 1 was analyzed for volume solids by the method outlined in section 2.2.1. That is, the volume solids determination was derived from an indirect measurement of wet volume and a direct measurement of dry film thickness and volume. For the paints under study, the density determined in Subtask 2.1 was used to calculate the wet volume of the paint. The procedure followed is given below.

Two film thickness measurement instruments were used. One is a dial guage micrometer as outlined in ASTM D 1005; the other instrument is an eddy current device.[11] These were used to measure the bottom thickness of the evaporating dishes. The thickness was determined by averaging readings from ten different spots in each dish. The approximate amount of paint was deposited into the dishes to give the desired final film thickness; efforts were made to spread the paint uniformly over the bottom of the dish by spinning at low speed. The paint was then allowed to dry or cure as appropriate. At the end of the curing schedule, the film thickness of the dry coating was measured with the two film measuring instruments with the measurements being made at the same positions in the dish without paint. The volume solids were then calculated based on the film thickness, area of the dish, wet paint density, and wet paint weight. Four determinations were made for each paint. The set of values were characterized by standard statistical techniques.

Subtask 2.3 Effect of Film Thickness on Volume Solids Values

The effect of film thickness on the volume solids values obtained was explored by determining the volume solids at two thickness levels, one greater than typically used and one less than typically used. This was done for one coating system from each generic type. The volume solids were measured by both the ASTM method and the film thickness method, as outlined in Subtasks 2.1 and 2.2. This task was undertaken since the film structure (pores, voids, trapped solvent) obtained can be dependent of the wet film thickness. This step was also necessary to equate the volume solids of the wet, deposited films in the laboratory to the realistic values of volume solids of the wet, deposited films spray applied in a shipyard.

Subtask 2.4. Calculated Volume Solids

The selected list of coatings from Task 1 all had the manufacturer's stated volume solids on their data sheets. These values were used as the calculated volume solids.

Task 3. Comparison of Methods

The volume solids data generated in Task 2 were compared and analyzed to assess the merits of the film thickness technique against the present ASTM method. The comparison was done by determining if the differences observed in volume solids were due to experimental error. The "student's t-test" was used.[12]

The t-test statistic was calculated by the expression

$$t_{\bar{d}} = \frac{(\bar{x}_1 - \bar{x}_2)}{\sqrt{S_{\bar{d}}}} \quad \text{with } (n_1 + n_2 - 2) \text{ degrees of freedom,}$$

where $\bar{x}_1$, $\bar{x}_2$ represent sample means, $S_{\bar{d}}$ = the pooled sample variance, and n_1 and n_2 are the sample sizes.

The probability associated with t was obtained from tables. The level of significance used for the comparisons was 0.05.

Task 4. Standardization of Volume Solids Measurement

In order to initiate standardization of the better method (Task 3) for determining volume solids in the marine industry, especially for coating suppliers, the results of the work, if warranted were to be presented to appropriate ASTM committees, such as F-25 Standards for Ship Building, D01.21.24 Volatile Content of Paint, and D-1.23.12 Film Thickness (dry), submitted for publication to the Journal of Coatings Technology, and presented at a Marine Coatings Conference. However, time and budget restraints have precluded this task.

2.4 Results

Each of the coatings selected in Task 1 were analyzed for volume solids by the ASTM D 2697-73 Method. Densities of the wet coatings were measured according to ASTM D 1475-60. Table 3 summarizes the volume solid values obtained. The values reported represent the average of at least four determinations.

The volume solids of the coatings selected in Task 1 were also determined by the film thickness technique as outlined in Section 2.2.1. The values are reported in Table 4. A sample calculation is given in Figure 2.

The effect of film thickness on the volume solids values was investigated by determining the volume solids at two additional thickness levels: one greater than recommended by the manufacturer and one less than recommended. Table 4 also contains the volume solids values for these samples.

Volume solids were also determined at different curing or conditioning schedules following the basic ASTM D 2697-73 procedure. Here, the temperature was varied. The results are listed in Table 5.

2.4.1 Discussion of the volume solids results obtained by using the ASTM

Coating: Sigma-Nucol CRHB-7311 (Chlorinated Rubber)

Procedure: Wet coating placed in drying pans and weight of wet paint determined. Coating spread over bottom of the pan then allowed to dry according to manufacturer's recommended conditions. Film thickness of dried film measured at eight locations distributed over the pan bottom using either an eddy current instrument or a dial gauge micrometer.

Drying Pan Area (A): 26.42 cm^2

Density of wet coating (ρ): 1.3987 $grams/cm^3$

	A	B	C	D
Weight of Wet Coating (W) (4 samples):	1.9798g	1.3364g	1.3508g	1.2709g
Average Dry Film Thickness (τ)	9.73 mil 0.0247cm	6.98 mil 0.0177cm	6.81 mil 0.0173cm	6.38 mil 0.0162cm
Volume Solids = $(A\tau\rho)/W$				
Calculated Volume Solids (%):	46.1	48.9	47.3	47.1

Figure 2. Example Volume Solids Determination by Film Thickness Method.

D 2697-73 method.

From Table 3, the volume solids value obtained using the ASTM D 2697-73 Method was larger than the manufacturer's value except the two ketimine-cured epoxies and one alkyd coating SIGMA-7240-7000. The experimental and reported volume solid values agreed for one coating, inorganic zinc-rich Matcote 1-289.

The method used by the various manufacturers to determine volume solids values is not known. However, it is surmised that the ASTM method was not the method used because of the very high percentage of differing results.

It is also noted from Table 3 that the volume solids values obtained using the manufacturer's recommended curing schedule are also larger than the manufacturer's stated volume solids values. Exceptions are, again, the two ketimine-cured epoxies, the alkyd coating Sigma-7240-7000, and an inorganic zinc-rich coating Matcote 1-289, where the values were the same.

For the standard ASTM Method, the precision of the method is good. In most cases, the magnitude of the 95 per cent confidence band (±1.96s) is less than one per cent absolute. However, there are cases when the precision is less. These are: Farboil #99PR (ketimine cured epoxy) with volume solids of 76.3 ± 2.2 per cent; Intertuf X8921/XV 1531 (waterborne) with volume solids of 54.9 ± 2.9 per cent; Matcote 1-289 (inorganic zinc-rich) with volume solids of 63.9 ± 2.9 per cent; Sigma MCF-7551 (inorganic zinc-rich) with volume solids of 74.7 ± 17.5 per cent. It is not known if these exceptions are indicative of specific problems in the applicability of the ASTM method to these materials or if additional experimentation would reduce the variance.

2.4.2 Discussion of the Volume Solids Results Obtained by Using the Film Thickness Measurement Method.

From Table 4, it is seen that the volume solids values determined at the manufacturer's recommended curing time (MRCT) and recommended film thickness (MRFT), are higher than the stated volume solids (MSVS) values in six of the seventeen cases and lower than the MSVS in nine cases.

In that portion of the study in which the film thickness was varied, it was found that lower film thicknesses than recommended gave lower volume solids values than those at the recommended thickness. For volume solids values determined at film thicknesses above the recommended thickness, values were higher than those obtained at MRFT. This indicates that in using the film thickness method, the possibility of trapping solvent or volatile material in heavier films could result in erroneous values.

2.4.3 Discussion of the Volume solids Values Based on Results Obtained by Altering the Curing Temperatures in ASTM D 2697-73.

As is shown in Table 5, the volume solids values obtained under the standard curing schedule, (3 Hrs. @ 105°C) are all larger than the MSVS in seven separate types of coatings. The volume solids values obtained using the MRCT are also larger than the MSVS. In six of the seven cases, the standard curing schedule temperature is lower than the MRCT. It is also shown in Table 5 that lower curing temperatures give higher volume solids values and higher temperatures give lower volume solids values. This trend was true even for conditioning to a constant weight at a given temperature.

2.4.4 Comparison of the ASTM D 2697-73 Method and the Film Thickness Method for Obtaining Volume Solids.

The results of the comparison between the ASTM method and the film thickness method for volume solids determination are presented in Table 6.

The comparison was made based on Student's t-test to quantitatively determine if the differences observed were due to experimental errors or not. In fifteen of the twenty-one paired comparisons, the disagreement in volume solids values could not be assigned to experimental or sampling error. Hence, the two methods do not, in general, give equivalent results.

The statistics indicate that the precision of the film thickness method is much less than the precision of the ASTM method. This lack of precision is primarily due to not being able to obtain a uniformly thick film. Inherent in the film thickness method is a requirement for constant film thickness.

During the course of the work on this project, several methods were tried to achieve uniform film thickness:

1. It was attempted to spread the wet paint uniformly over the bottom of the dishes by spinning them at various speeds. The apparatus used is depicted in Figure 3.

The spinning apparatus was made from a small laboratory stirrer. An aluminum dish was cemented to the shaft of the stirrer. This dish served as a holder for the dishes containing the wet paint samples. A rheostat was connected to the stirrer in order to provide variable rotation speeds.

2. The aluminum dishes were also rotated very slowly manually and placed on a level surface to cure.

3. The viscosity of the paint was lowered with appropriate solvent and methods 1 and 2 above tried.

4. Aluminum dishes containing wet paint were placed in an ultrasonic bath containing water to allow the vibrations to produce a uniform film.

5. Method 4 was also attempted using liquids denser than water, e.g., trichloro-trifluoroethane and methylene chloride. Liquids lighter than water were also used, e.g., mineral spirits and VM & P naphtha.

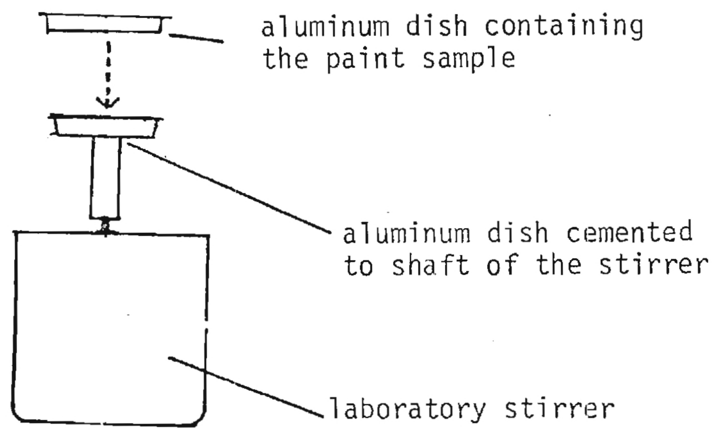

Figure 3. Diagram of spinning apparatus

All of the above methods failed to consistently produce sufficiently uniform film thicknesses to give the film thickness method the precision of the ASTM method.

Lastly, under equivalent curing or conditioning schedules, there was no savings of time or sample handling with the film thickness method over the ASTM method. In fact, in many instances, the time was actually longer.

TABLE 3

VOLUME SOLIDS AS DETERMINED BY

THE ASTM D 2697-73 STANDARD METHOD

PAINT	*MSVS	ASTM D 2697-73 3 hrs @ 105°C	**MRCT
I. High-Build Ketimine Cured Epoxy			
1. Farboil #99	87.0	73.5	76.7
2. Farboil #99 PR	87.0	76.3	85.0
II. Amine and Amine Adduct Cured Epoxy			
1. Carboline - 187HFP	66.0±1	68.6	73.8
2. Napko - 7-2371	44.3	55.6	57.8
3. Sigma - EHB - 7433	70.0	76.6	80.8
III. Polyamide Cured Epoxy			
1. Deco-Rez-DE-3490	40.0	44.3	45.2
2. Matcote Co. - 1-844	50.0±2	63.7	67.2
3. Carboline - 193	50.0±1	56.4	57.3
IV. Vinyl Coatings			
1. Imperial Co.-#321 C-Flex	28.0±1	42.4	46.3
2. Sigma-NUCOL #7352	24.0	24.2	26.1
V. Chlorinated Rubber			
1. Carboline-3631HB	34.0±1	39.3	44.9
2. Sigma NUCOL-7311	47.0±1	49.9	52.6
VI. Urethanes			
1. Carboline - 132	55.0±1	56.3	55.8
2. Imperial--#1001	46.0	54.3	56.6
VII. Water-base Coatings			
1. International Intertuf-X8921/XV1531	53.5	55.9	53.8
2. Sigma-7445	39.0	52.9	53.6
3. Porter-Epoxy 6610	36.4±2	41.7	42.7

*MSVS - Manufacturer's Stated Volume Solids
**MRCT - Manufacturer's Recommended Curing Time

TABLE 3

VOLUME SOLIDS AS DETERMINED BY

THE ASTM D 2697-73 STANDARD METHOD (Cont'd)

PAINT		*MSVS	ASTM D 2697-73 3 hrs @ 105°C	**MRCT
VIII.	Alkyd Coatings			
1.	Matecote-2400	35.0±2	44.7	46.3
2.	Sigma - 7240-7000	50.0	41.0	44.1
IX.	Inorganic Zinc-rich Coatings			
1.	Matcote 1-289	63.9	63.9	63.9
2.	Sigma MCF-7551	56.0	74.7	74.2

*MSVS - Manufacturer's Stated Volume Solids
**MRCT - Manufacturer's Recommended Curing Time

TABLE 4

VOLUME SOLIDS DETERMINED BY THE FILM THICKNESS MEASUREMENT METHOD

Coatings	*MSVS	**MRFT	DIAL-GUAGE MICROMETER						****"DUAL-SCOPE" MODEL AE					
			VOLUME SOLIDS	*** D.F.T. MRFT	VOLUME SOLIDS	D.F.T. below MRFT	VOLUME SOLIDS	D.F.T. above MRFT	VOLUME SOLIDS	*** D.F.T. MRFT	VOLUME SOLIDS	D.F.T. below MRFT	VOLUME SOLIDS	D.F.T. above MRFT
I. High-Build Ketimine-Cured Epoxy														
1. Farboil #99E	87.0	4-6	61.1	6.2	65.6	3.3	59.5	7.1			68.4	3.5		
2. Farboil #99 PR	87.0	4-6	67.4	4.8			66.7	6.5	67.0	4.8			68.4	7.6
II. Amine and Amine Adduct Cured Epoxy														
1. Carboline-187 HFP	66.0	4-8	78.1	7.6			78.8	8.4						
2. NAPKO-7-2371	44.3	2-4	43.3	3.0	40.9	1.9	45.6	4.1	41.6	2.9	40.1	1.9	43.2	3.9
3. SIGMA - EHB-7433	70.0	10.0	75.8	10.2	67.3	5.6	82.0	13.9			65.1	5.3		
III. Polyamide Cured Epoxy														
1. Deco-Rez-DE-3490	40.0	2-5	40.0	3.6	28.1	2.0	46.0	6.8	41.2	3.6	31.7	2.0		
2. Matcote Co.-1-844	50.0	2-3					57.1	3.4						
3. Carboline-193	50.0	3-4	56.4	3.2			66.5	4.1			–			
IV. Vinyl Coatings														
1. Imperial - C-Flex #321	28.0	3-4	59.8	3.5	28.6	1.6	57.6	5.4	63.0	3.6	48.4	1.5	58.7	4.4
2. SIGMA-NUCOL #7352	24.0	3.0	24.2	3.3	21.0	2.3	26.2	3.9	28.3	3.8	23.2	2.4	29.3	4.4
V. Chlorinated Rubber														
1. Carboline-3631HB	34.0	3-0			22.0	2.3								
2. SIGMA-NUCOL - 7311	47.0	3-4	44.5	3.8	44.8	2.2	48.6	6.6	47.8	3.9	45.6	2.4	50.4	6.6
VI. Urethanes														
1. Carboline-132	55.0	1.5-2.0	44.8	1.9			45.8	2.3						
2. Imperial-#1001	46.0	2-3	45.8	2.5	36.7	1.4	48.1	4.4	52.5	2.9	38.1	1.4	46.1	4.2

* MSVS - Manufacturer's stated volume solids (%)
** MRFT - Manufacturer's recommended film thickness (Mils)
*** D.F.T. MRFT-Dry film thickness in the manufacturers recommended film thickness range (Mils)
**** Eddy - Current film thickness measuring instrument-(Maximum thickness Measurements-8 Mils)

TABLE 4

VOLUME SOLIDS DETERMINED BY THE FILM THICKNESS MEASUREMENT METHOD (Cont'd)

Coatings	*MSVS	**MRFT	DIAL-GUAGE MICROMETER						****"DUAL-SCOPE" MODEL AE					
			VOLUME SOLIDS	*** D.F.T. MRFT	VOLUME SOLIDS	D.F.T. below MRFT	VOLUME SOLIDS	D.F.T. above MRFT	VOLUME SOLIDS	*** D.F.T. MRFT	VOLUME SOLIDS	D.F.T below MRFT	VOLUME SOLIDS	D.F.T. above MRFT
VII. Water-based Coatings														
1. International-Intertuf X8921/XV1531	53.5	7-14	48.9	9.5	45.4	5.6					53.1	6.5		
2. Sigma-7445	39.0	3.2	51.2	3.3	50.0	2.4	54.3	4.6	46.8	3.3	47.0	2.2	51.8	4.5
3. Porter-Aqualock-6610	36.4	2.5	34.6	2.6	32.3	1.8	34.6	5.0	33.1	2.6	30.8	1.9	34.0	3.6
VIII. ALKYD COATINGS														
1. Matecote Alkyd-2-400	35.0	1.5	28.7	1.6	25.1	1.4	33.6	1.8						
2. Sigma-7240-7000	50.0	1.5	25.6	1.5	24.5	1.4	30.1	2.6	24.0	1.6	25.3	1.4	33.5	2.9
IX. Inorganic Zinc-rich Coatings														
1. Matecote - 1-289	63.9	3-5					70.5	5.9						
2. Sigma-MCF-7551F	56.0	2.0					74.5	3.9						

* MSVS - Manufacturer's stated volume solids (%)
** MRFT - Manufacturer's recommended film thickness (Mils)
*** D.F.T. MRFT-Dry film thickness in the manufacturers recommended film thickness range (Mils)
**** Eddy - current film thickness measuring instrument-(Maximum thickness Measurements-8 Mils)

TABLE 5

COMPARISON OF VOLUME SOLIDS RESULTS
BY ALTERING THE ASTM D 2697-73 METHOD
CURING TEMPERATURES

PAINT	* MSVS	** MRCT	*** MRCT-VS	VOLUME SOLIDS AT STATED TEMPERATURES				
				3 Hrs. @ 221°F 105°C	**** 150°F 65.5°C	**** 200°F 93.3°C	**** 248°F 120°C	**** 303°F 150°C
CHLORINATED RUBBER								
Carboline-3631 HB	34±1	5 Hrs. 75°F (24°C)	44.9	39.3	40.1	38.1		
Sigma-NUCOL 7311	47±1	8 Hrs. 68°F (20°C)	52.6	49.9		53.5		
URETHANE								
Carboline 132	55±1	5 days 90°F (32°C)	55.8	56.3	53.1	48.8		
AMINE ADDUCT CURED EPOXY								
Napko 7-2371	44.3	2 days 75°F (24°C)	57.8	55.6			49.9	46.0
AMINE CURED EPOXY								
Carboline-187 HFP	66±1	20 Hrs. 150°F (66°C)	73.8	68.6	71.7	70.7		
POLYAMIDE CURED EPOXY								
Deco-Rez-DE-3490	40.0	12-14 Hrs. 77°F (25°C)	45.2	44.3			38.5	38.1
Matcote 1-844	50±2	18 Hrs. 77°F (25°C)	67.2	63.7	61.4	60.5		

*MSVS-Manufacturer's Stated Volume Solids
**MRCT-Manufacturer's recommended Curing Temperature
***MRCT-VS-Manufacturer's Recommended Curing Temperature-Volume Solids
**** - Samples heated until constant weight obtained.

TABLE 6

Student's t-Test of Statistical Significance Between
ASTM D 2697-73 and Film Thickness Method

PAINT	$\bar{X}_1 \pm 1.965$ ASTM Method V.S. (Sample Means)	$\bar{X}_2 \pm 1.965$ F.T.M. V.S. (Sample Means)	n_1 Sample Size ASTM Method	n_2 Sample Size F.T.M.	S_1^2 Variance ASTM Method	S_2^2 Variance F.T.M.	S^2 Pooled Estimate Variance	$S_{\bar{d}}$ Sample Variance for Diff. in sample means	$t_{\bar{d}}$ Comparison of Means	Pr(t) Probability/t	CONCLUSION
KETIMINE CURED EPOXY											
1. Farboil #99E	73.5±2	61.1± 9.8	4	4	.010	25.06	25.06	2.50	4.96	.002	Difference Due to Method
2. Farboil #99PR	76.3±2.2	66.7± 28.6	4	4	1.300	212.20	106.75	7.31	1.32	.241	Sampling Difference
AMINE & AMINE ADDUCT CURED EPOXY											
3. Carboline 187HFP	68.6±2	78.1± 2	4	2	.016	1.05	.27	.45	21.16	.000	Difference Due to Method
4. NAPKO 7-2371	55.6±.4	43.3 ± 5.6	4	4	.046	8.19	4.12	1.44	8.57	.000	Difference Due to Method
5. Sigma EHB-7433	76.6±3	75.8 ± 6.7	4	4	.017	45.51	22.76	3.37	.24	.848	Sampling Difference
POLYAMIDE CURED EPOXY											
6. Deco-Rez-DE-3490	44.3±.1	40.0±5	4	4	.003	25.61	12.81	2.53	2.06	.086	Sampling Difference
7. Matcote Co.-1-844	63.7±.2	57.1±3	4	3	.011	8.86	3.55	1.44	4.58	.004	Difference Due to Method
8. Carboline-193	56.4±.5	56.4 ± 6.7	4	2	.065	11.80	3.00	1.20	0.00	1.000	Sampling Difference
VINYL COATINGS											
9. Imperial Co. G-Flex	42.4±.37	28.6 ± 2.7	4	3	.035	1.88	1.16	.82	16.80	.000	Difference Due to Method
10. Sigma-NUCOL #7352	24.2±.26	23±4.8	4	3	.018	1.96	2.39	1.18	3.81	.009	Difference Due to Method
CHLORINATED RUBBER											
11. Carboline 3631HB	39.3±.36	21.3 ± 4.4	4	4	.033	5.15	2.59	1.14	15.82	.000	Difference Due to Method
12. Sigma NUCOL-7311	49.9±.09	47.4 ± 2.4	4	4	.002	1.44	.96	.69	3.61	.011	Difference Due to Method

* V.S. - Volume Solids
** F.T.M. - Film Thickness Method

TABLE 6 (Continued)

Student's t-Test of Statistical Significance Between
ASTM D 2697-73 and Film Thickness Method

PAINT	$X_1 \pm 1.96s$ ASTM Method Volume Solids Sample Means	$X_2 \pm 1.96s$ F.T.M. Volume Solids	n_1 Sample Size ASTM Method	n_2 Sample Size F.T.M.	S_1^2 Variance ASTM Method	S_2^2 Variance F.T.M.	S^2 Pooled Estimate Variance	$S_{\bar{d}}$ Sample Variance for Diff. in sample means	$t_{\bar{d}}$ Comparison of Means	PR(t) Probability/t	CONCLUSION
URETHANES											
13. Carboline-132	56.3±.5	51.5 ± 6.6	4	3	.073	11.21	4.53	1.63	2.95	.024	Difference Due to Method
14. Imperial-#1001	54.3±.2	47.0 ± 9.1	4	4	.013	21.67	10.84	2.32	6.29	.000	Difference Due to Method
WATER-BASE COATINGS											
15. International Intertuf X8921/XV1531	54.9±2.9	48.9 ± 2.8	4	4	2.168	2.09	2.13	1.03	5.83	.000	Difference Due to Method
16. Sigma-7445	52.9±3.9	50.4 ± 7.6	4	4	3.953	14.93	9.44	2.17	1.52	.184	Sampling Difference
17. Porter Epoxy 6610	41.7±37	34.2 ± 2.6	4	4	.036	1.70	.87	.66	11.38	.000	Difference Due to Method
ALKYD COATINGS											
18. Matecote-2400	44.7±.3	33.6 ± 5.3	4	3	.035	7.29	2.94	1.31	8.49	.000	Difference Due to Method
19. Sigma-7240-7000	41.0±.2	33.1 ± 6.6	4	3	.015	11.33	4.54	1.63	10.61	.000	Difference Due to Method
INORGANIC ZINC-RICH COATINGS											
20. Matecote 1-289	63.0±.9	70.5± 8.7	4	3	2.130	19.77	9.19	2.32	2.84	.036	Difference Due to Method
21. Sigma MCF-7551	74.7±17.5	74.5± 11.3	4	2	79.710	33.06	96.24	8.50	.02	1.000	Sampling Difference

* F.T.M. - Film Thickness Method

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